IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Appl. No.	:	**10/628,424**	Confirmation No.: **5300**
Applicant	:	**Read**	
Filed	:	**07/29/2003**	
TC/A.U.	:	**1745**	
Examiner	:	**Jane J. Rhee**	
Docket	:	**ARL 01-37**	
Customer No.	:	**37064 Office of Command Counsel** **U.S. Army Materiel Command**	

For: **Electrolyte for Metal-Oxygen Battery and Method for Its Preparation**

DECLARATION OF JEFFREY A. READ OF PRIOR INVENTION IN THE UNITED STATES TO OVERCOME A CITED REFERENCE UNDER 37 CFR §1.131

I, Jeffrey A. Read, declare as follows:

1. I am the sole inventor of the invention disclosed in the above-identified application for patent.

2. This declaration is to establish completion of the invention being claimed in the above-referenced application in the United States at a date prior to October 5, 2001, which is the effective date of U.S. Patent Application Publication US 2004/0091774 A1 (Narang, *et al.*) that was cited in the Final Office Action, mailed August 30, 2006.

3. I understand that pending claims 13-17 of the pending application have been rejected under 35 U.S.C. §102(e) as being anticipated by U.S. Patent Application Publication US 2004/0091774 A1 (Narang, *et al.*). I further understand that while U.S. Patent Application Publication US 2004/0091774 A1 (Narang, *et al.*) was Filed on 4 October 2002, it claims priority to U.S. Provisional Application Number 60/327,468, which was Filed on 5 October 2001.

4. I state that I have worked extensively in the area lithium-ion and lithium-air batteries for over 7 years in my current position within the Sensors and Electron Devices Directorate, Directed Energy Branch, Army Research Laboratory (ARL). I believe my invention was reduced to practice prior to the filing date of Narang, *et al.* on 5 October 2001. As evidence of my reduction to practice prior to 5 October 2001, attached at Appendix A is a copy of the Invention Disclosure submitted to the ARL Legal office on 2 May 2001. As further evidence of my reduction to practice I am submitting copies of pages 89, 91-95, 97-98, and 100 of notebook number 3 (No. 8830) and pages 1-8, 11-28, and 32-34 of notebook number 4 (No. 8115), which are attached as Exhibit B.

5. I state that the above-referenced application was filed on my behalf on July 29, 2003, and that I had no control over the processing, and Filing of the Application, which was under the control of the ARL Legal Office and the Center for Patent Prosecution Excellence at Headquarters, U.S. Army Materiel Command (AMC), Fort Belvoir, Virginia, after I submitted the Invention disclosure to the Legal Office. I exercised due diligence in submitting an invention disclosure and causing the above-referenced patent application to be filed, and, to the best of my knowledge, due diligence was exercised by the Legal Office, Headquarters AMC, and the law firm contracted with to prepare a Draft Patent Application for submission to Headquarters AMC for review and Filing.

7. I declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true. These statements are made with the knowledge that willful false statements and the like so made are punishable by fine or

imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

/s/Jeffrey A. Read_____ Date:___02/28/2007___
Jeffrey A. Read

01-37

Army Research Laboratory Invention Disclosure

Instructions: Complete the below items, print a hard copy, sign, date, and send to the Intellectual Property Law Division of ARL (AMSRL-CS-CC-IP) (301-394-3790) (301-394-3972 FAX)

INVENTION TITLE: Electrolytes for Lithium-Air Cell

INVENTORS:

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Zip:

4th Name:
Street Address:
City:
State:
Zip:

INVENTION HISTORY:

a) DATE of Conception of the Invention: March 27, 2001
b) PLACE: Army Research Center, Adelphi Laboratory Center, Adelphi, MD
c) DATE of First Sketch/Drawing:
d) PLACE:
e) DATE of First Written Description of Invention: March 30, 2001
f) PLACE: Army Research Center, Adelphi Laboratory Center, Adelphi, MD
g) DATE of First Disclosure to Others: April 27, 2001
h) PLACE: Army Research Center, Adelphi Laboratory Center, Adelphi, MD
i) DATE of Completion of Model (if any):
j) PLACE:
k) DATE of Completion of Full Scale Item: April 5, 2001
l) PLACE: Army Research Center, Adelphi Laboratory Center, Adelphi, MD
m) DATE of First Test of Invention: April 9, 2001
n) PLACE: Army Research Center, Adelphi Laboratory Center, Adelphi, MD
o) RESULTS of First Test: 1M $LiPF_6$ EC:DMC > 1M $LiPF_6$ γ-Butyrolactone > 1M $LiPF_6$ Propylene Carbonate

LIST INDIVIDUALS HAVING FIRST HAND KNOWLEDGE OF THE INVENTION HISTORY:
List their names, address and the features of the invention they have knowledge of
a)
b)
c)
d)

LABORATORY NOTEBOOK DATA:
List the lab notebook number and pages where the invention is described
Notebook #3 (No. 8830), p.89, 91-95, 97-98, 100.
Notebook #4 (No. 8115), p. 1-8, 11-28, 32-34.

PUBLICATION OF THE INVENTION:
If a description of the invention has been published, list the type of publication and the dates. Also, identify any further planned reports or publications. If none, so state. **None**

LIST ANY KNOWN RELATED PATENTS, PUBLICATIONS or PATENT APPLICATIONS:
Also identify any previous reports, drawings, publications, or correspondence describing or showing the invention. List any known closely related patents, patent applications, reports, publications, devices, or methods. If none, so state.

- K.M. Abraham and Z. Jiang, US Patent 5,510,209
- K.M. Abraham and Z. Jiang, *J. Electrochem. Soc.*, **143** (1996) p.1

IS AN EMBODIMENT OF THE INVENTION AVAILABLE FOR INSPECTION? Yes
If so, where? Army Research Center, Adelphi Laboratory Center, Adelphi, MD

NATURE AND EXTENT OF PAST USE, PRESENT USE, AND FUTURE USE:

Past: **None**

Present: Laboratory Cells

Future: Batteries for Military and Commercial Applications

DESCRIPTION OF THE INVENTION:
Provide the following information concerning the disclosed invention and in the indicated sequence:

A. Specifically describe the invention and its operation. You may use and attach copies of sketches, prints, photographs, paper, and illustrations, which should be signed, witnessed and dated. Use numbers and descriptive names in descriptions and drawings. For inventions that are methods list the steps involved in the method. For inventions that are apparatus describe all the elements.

The invention is a series of electrolytes and electrolyte solvents used in an electrochemical cell where the cathode has access to oxygen from the air or other source. Additionally, the invention is a method of choosing electrolytes and electrolyte solvents used in an electrochemical cell where the cathode has access to oxygen from the air or other source.

Propylene carbonate(PC), γ-butyrolactone(g-BL), ethylene carbonate(EC), dimethyl carbonate (DMC), 1,2-dimethoxyethane (DME), tetrahydrofuran (THF), and

tetrahyropyran (THP) were used individually or in combination to prepare electrolyte mixtures with $LiPF_6$ salt. **Figure 1** compares the voltage versus capacity curves of lithium-air cells at $0.2mA/cm^2$ with 1M $LiPF_6$ electrolytes made from these of solvents. **Figure 2** compares the specific capacity of a series of lithium-air cells at 0.05, 0.2 and $1.0mA/cm^2$ with this same series of electrolytes.

From **figures 1** and **2** it can be observed that the discharge capacity and rate capability of the lithium air cell is directly related to the electrolyte used. By comparing the solubility of oxygen in these solvent mixtures

$$PC:THF \cong PC:THP > g\text{-}BL > PC$$

to the discharge capacity and rate capability of the lithium air cells

$$PC:THF > PC:THP > g\text{-}BL > PC$$

it is observed that the ability of the electrolyte to dissolve oxygen is directly related to the performance of the lithium air cell. The solubility of oxygen in EC:DMC and PC:DME is not known at this time. Higher oxygen solubility leads to higher discharge capacity and rate capability. By choosing solvents and salts that improve the solubility of oxygen in the electrolyte, the capacity and rate capability of the lithium-air cell can be improved. The solvents and salts that can be chosen are not limited to the ones mentioned in this disclosure but could include solvents such as perfluorobutylperfluorotetrahydrofuran (FC-80) which is known to have high oxygen solubility. Various salts and additives could also be used to improve oxygen solubility.

The lithium-air cell operates based on the principle that the air cathode (composed of a catalytic material such as a carbon black: Super P, Vulcan XC-72, or Acetylene Black; or other catalytic material such as MnO_2), reduces oxygen from the air in an organic electrolyte based electrochemical cell. The catalytic material in the air electrode reduces O_2 to O_2^{-2} or O^{-2}. The reduced oxygen then reacts with lithium to form Li_2O_2 or Li_2O that deposits on the surface and in the pores of the air electrode. The operating voltage for such a cell is 2.0-2.8V, while the open circuit voltage is 2.85V. The catalytic material provides numerous sites for the deposition of Li_2O_2 or Li_2O due to a large surface area.

B. State the advantages of the invention over presently known devices, systems or processes. Also discuss/provide a background of the prior art.

Metal-Air batteries using aqueous electrolytes are well known with Iron/air, Zinc/air and Aluminum/air being the most studied. The zinc/air battery has been commercialized for hearing aid devices and pagers. Abraham and Jiang[1,2] recently described a lithium-air battery using organic electrolyte. This battery utilizes a carbon cathode (graphite, acetylene black) that reduces oxygen to form Li_2O_2 or Li_2O as described above.

The lithium-air cell operates based on the principle that the air cathode (composed of a catalytic material such as a carbon black: Super P, Vulcan XC-72, or Acetylene Black; or other catalytic material such as MnO_2), reduces oxygen from the air in an organic electrolyte based electrochemical cell. The catalytic material in the air electrode reduces O_2 to O_2^{-2} or O^{-2}. The reduced oxygen then reacts with lithium to form Li_2O_2 or Li_2O that deposits on the surface and in the pores of the air electrode. The operating voltage for such a cell is 2.0-2.8V,

while the open circuit voltage is 2.85V. The catalytic material provides numerous sites for the deposition of Li_2O_2 or Li_2O due to a large surface area.

The advantage of this invention over presently known devices is that the capacity and rate capability of the presently known devices can be improved by choice of electrolytes and electrolyte solvents. The discharge capacity and rate capability are directly related to the ability of the electrolyte solvent to dissolve oxygen. By properly choosing the electrolyte solvents from the list above or from any list of solvents known to be stable in an organic electrolyte based lithium-air cell, the discharge capacity and rate capability of the lithium air cell can be improved.

C. Discuss the problems which the invention is designed to solve, referring to any prior invention of a similar nature with which you may be familiar.

The invention is designed to solve the problem of providing more energy to portable devices. Storing more capacity in less weight is a desirable property of any new electrochemical system. This invention succeeds in providing more capacity and better rate capability.

D. List all known and other possible uses for the invention. **None**

E. List the features of the invention that are believed to be novel.

1) The invention provides a series of electrolytes that improve the capacity and rate capability of the organic electrolyte based lithium air cell.

2) The invention provides a method of choosing electrolytes that improve the capacity and rate capability of the organic electrolyte based lithium air cell.

SIGNATURE OF ALL INVENTORS:
All inventors must sign and date this document.

SIGNATURE: Jeffrey Read DATE: 05/02/01

ORGANIZATION: ARL

SIGNATURE: _____ DATE: _____

ORGANIZATION: _____

SIGNATURE: _____ DATE: _____

ORGANIZATION: _____

RIGHTS IN INVENTIONS MADE BY GOVERNMENT EMPLOYEES

The Government shall obtain the entire domestic right, title and interest

in and to any invention made by any Government employee:

a) During working hours, or

b) With a contribution by the Government of facilities, equipment, materials, funds or information, or of time or services of other Government employees on official duty, or

c) Which bears a direct relation to or is made in consequence of the official duties of the inventor.

When you report your invention to the Intellectual Property Law Branch, you will be asked to sign a statement that you have read Executive Order 10096, 37 CFR 501, and AR 27-60 which discuss rights in inventions and the appeal process. You will also be asked to sign DA FORM 2871-R entitled Invention Right Questionnaire in which you will indicate either a desire to assign the invention to the Government, or to ask for a rights determination.(A short version of this form is available on the legal office web site)

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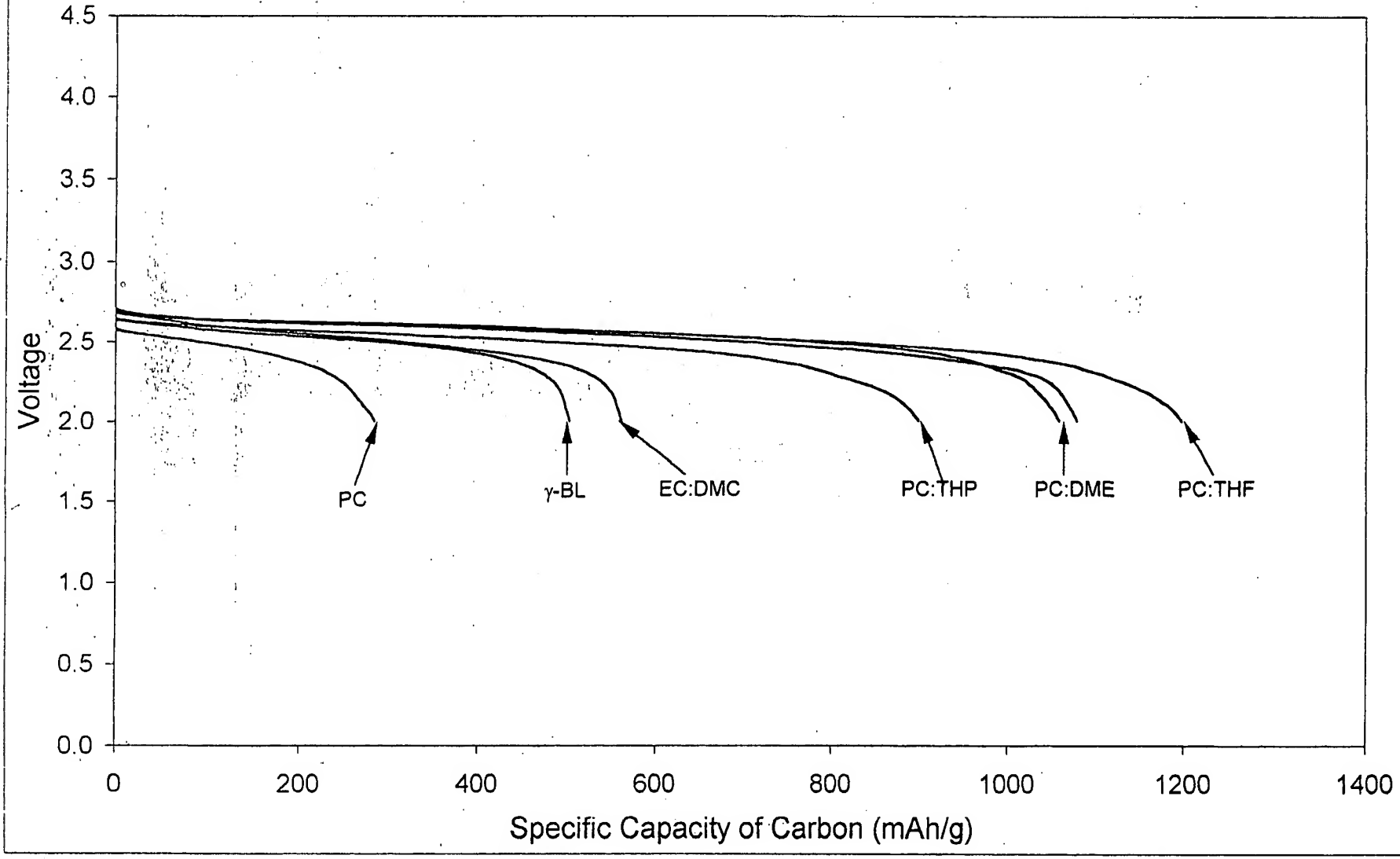
[1] K.M. Abraham and Z. Jiang, J. Electrochem. Soc., 143 (1996) 1

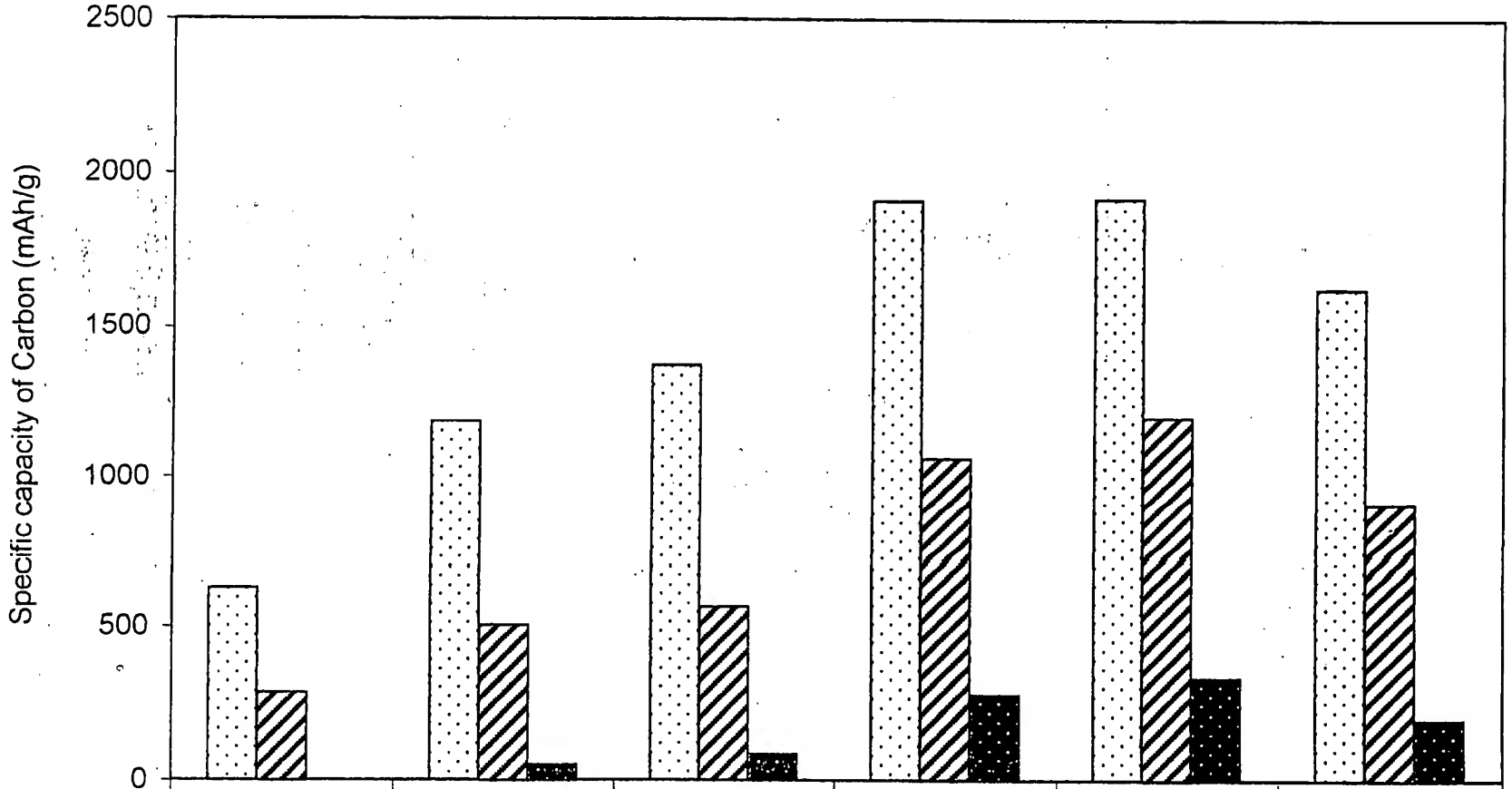
[2] US Patent 5,510,209

Figure 1
(Specific Capacity of Carbon @ 0.2 mA/cm^2)



5/02/01

Figure 2



	PC	g-BL	EC:DMC	PC:DME	PC:THF	PC:THP
0.05 mA/cm2	626	1184	1369	1906	1911	1618
0.2 mA/cm2	284	503	562	1059	1198	899
1.0 mA/cm2	0	51	86	273	328	192

APPENDIX A 7/7

5/02/01

EXAIBIT B 1/38

Lithium - Air Cathodes 12/27/00

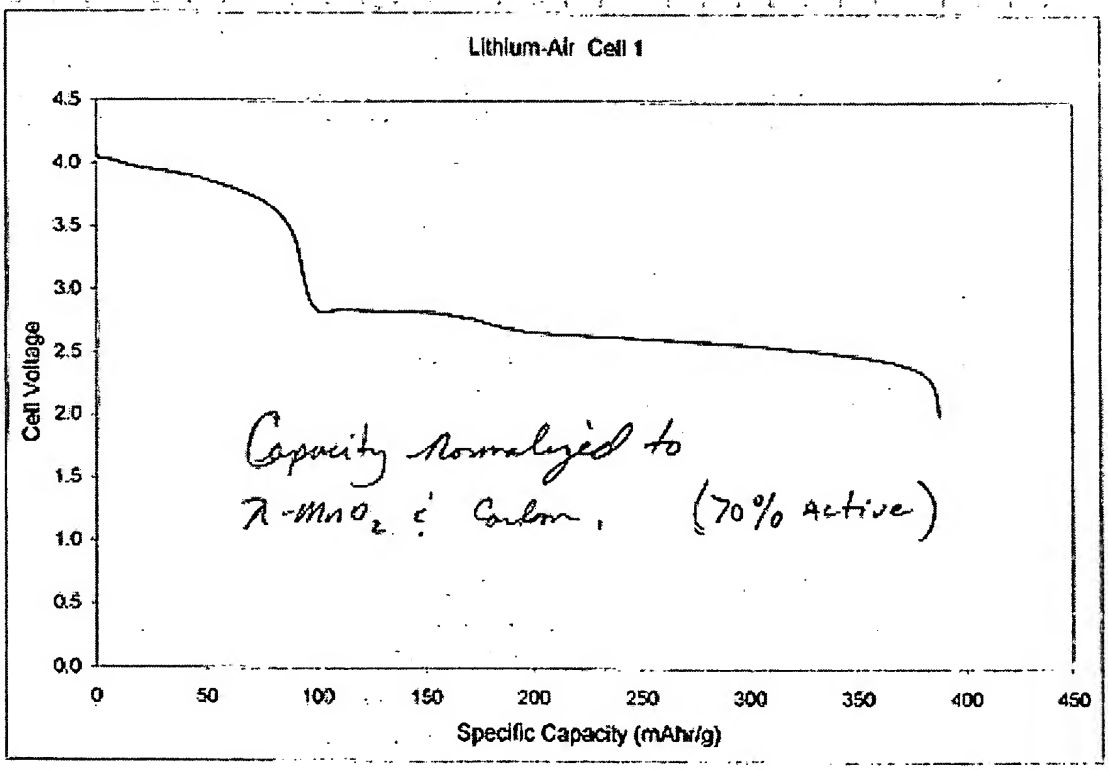
Initial Test Cell - 50% λ-MnO_2, 20% Super P, 30% Kynar 2801

Capacity = 21.9 mAh.

Cathode wt ≈ .0806 g (70% Active)

→ When disassembled, the cathode appeared to have circles on the lithium side of the cathode where the air holes were situated on the back side of the cathode. The cathode was hard in this area when examined, while areas not exposed to air holes were soft.

→ Cell: Li°, Celgard 2300, 1M $LiPF_6$ PC/DME (1:1), Air cathode on Al Grid, O_2 (pure) in the gauch.



Jeff Read
ARL

Li-O2.xls
12/27/00

Continued on Page _____

Signature

12/22/00
Date

Read and Understood Date Read and Understood Date

EXHIBIT B 2138

Lithium - Air Cathode

12/28/00

λ-MnO_2 (AW-46)	-25.0%
Super P	-15.0%
Kynar 2801	-20.0%
DBP	-40.0%

λ-MnO_2 (AW-46)	-8.4538 g
Super P	-5.0713 g
Kynar 2801	-6.7282 g
DBP	-13.4261 g

Acetone -102.44 g + 10g

gap = 1.194 mm

Lot#: N3 P91A

Initial formula

λ-MnO_2 (AW-46)	= 25%
Super P	= 15%
Kynar 2801	= 20%
DBP	= 40% ← Increase to 70%

λ-MnO_2 (AW-46)	= 12.5%
Super P	= 7.5%
Kynar 2801	= 10.0%
DBP	= 70%

λ-MnO_2	= 8.3261 g
Super P	= 5.1085 g
Kynar 2801	= 6.7095 g
DBP	= 46.5779 g

Acetone = 73.5 g + 42.8 g - 10.2 g = 106.1 g

Gap 1 = 1.194 mm

Gap 2 = 2.286 mm

Lot # N3 P91B

Continued on Page _____

Signature

12/28/00

Date

Read and Understood | Date | Read and Understood | Date

Lithium - Air Cathodes

12/28/00

λ-MnO_2 = 18.75%
Super P = 11.25%
Kynar 2801 = 15%
DBP = 55%

λ-MnO_2	= 8.3101 g	(Aw-46)	(18.64%)	> 30.10%
Super P	= ~~5.0881 g~~ (AN)	5.1077 g	(11.46%)	
Kynar 2801	= 6.8363 g		(15.33%)	
DBP	= 24.3324		(54.57%)	

1/2/00

44.5865 g total

Acetone = 82.5 g + 7.7 g

Cast @ 1.184 mm & 2.286 mm

Dried to 10 mils & 24 mils

Lot # N3P92A

Cell Build - (5 cm² cell)

Cell #	Cath wt.	OCV	Before dis. Imp @ 10KHz	→ Before wt. in air	wt. in water (1/2/01, ~~19.8°C~~)	wt. in water (1/3/01, 19.8°C, 761 mmHg)	wt. in water (1/8/01, 21.2°C, 749 mmHg)	wt. in water (1/1[…], 22.[…], 75[…])
LA 1	.1704 g	4.14 V	~~34 Ω~~ 67 Ω	14.82 g	-~~46.39~~ g	-46.22 g	-37.50	-34.[…]
LA 2	.3641 g	Short						
LA 3	.7266	4.13 V	34 Ω	15.34 g	-~~87.07~~ g	-56.81 g	~~[illegible]~~	-11.[…]

Cathode Lot # N3P92A
LAM'd @ 300F, 1 pass
Extract 3X in MeOH for 30 min
Dried under vacuum 2 hrs. 2:00 - 4:00 pm
Activated w/ 8 ml of 1M $LiPF_6$ PC:DME (Lot # N3P84B)
Measured impedance after overnight rest. 1/3/01, Filled bag w/ O_2
Put on to discharge at .1 mA/cm² to 2V. PCXIR [illegible] Test.
Measured O_2 volume change.

Continued on Page ______

[signature] Signature 1/16/01 Date

Read and Understood ______ Date ______ Read and Understood ______ Date ______

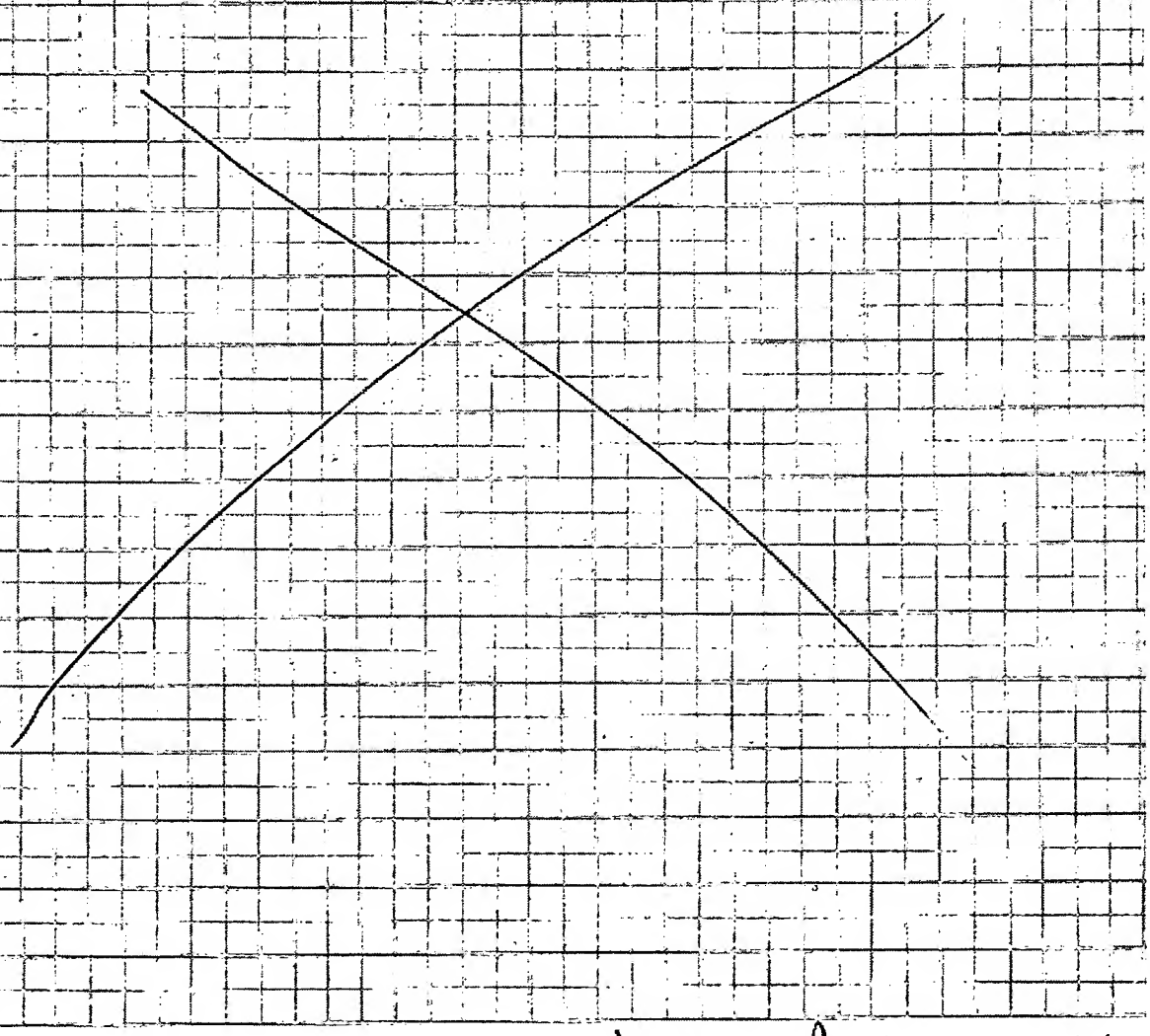
O_2 volume vs. mAh Capacity 1/4/01

4e⁻ - Assume 4e⁻ reaction for reduction of O_2. ($4Li + O_2 \rightarrow 2Li_2O$)

$$\frac{4 \text{ moles } e^-}{\text{mole } O_2} \times \frac{96500 \text{ C}}{\text{mole } e^-} \times \frac{1 \text{ mole } O_2}{22.4 \text{ L}} \times \frac{1 \text{ liter}}{1000 \text{ mL}} \times \frac{1 \text{ A}}{1 \text{ C/s}} \times \frac{1 \text{ hr}}{3600 \text{ s}} \times \frac{1000 \text{ mA}}{\text{A}}$$

2e⁻ - Assume 2e⁻ reaction for reduction of O_2 ($2Li + O_2 \rightarrow Li_2O_2$)

- $\frac{2.40 \text{ mAh}}{\text{mL}}$ or $\frac{.418 \text{ mL}}{\text{mAh}}$



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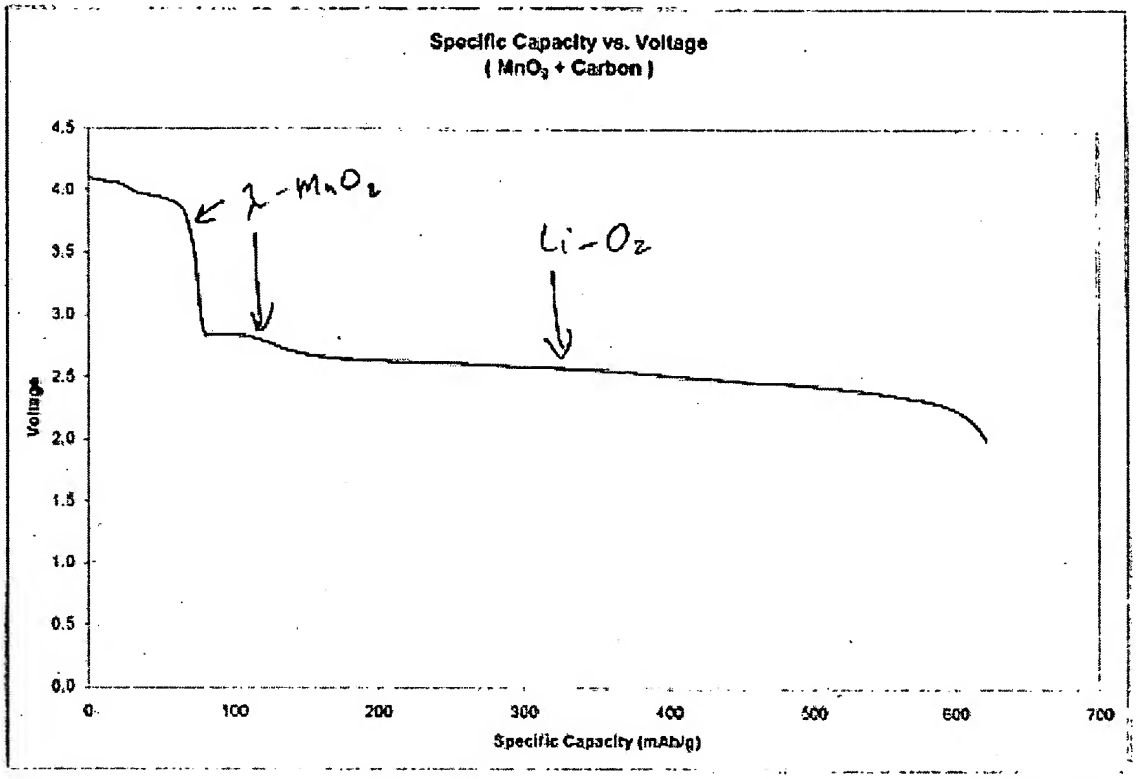
Signature 1/4/01 Date

Read and Understood Date Read and Understood Date

Discharge Profiles of Lithium-Air cell (LA1) 1/8/01



Cell discharged at 1mA/cm² to 2V.
Active weight = .0513 g
Total capacity = 31.93 mAh

The voltage curve shows both the λ-MnO_2 discharge profile and the $Li-O_2$ discharge voltage profile.

The gas volume change associated with the discharge was indicative of the formation of Li_2O_2, as shown on the next page. The OCV measured 3 days at the end of discharge seems to indicate that the primary discharge product is Li_2O_2.

Continued on Page _____

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Signature

1/8/01
Date

Read and Understood | Date | Read and Understood | Date

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Calculation of 2e⁻ or 4e⁻ discharge reaction. 1/8/

The volume measurements on LA1 were used to determine whether Li_2O_2 or Li_2O were being formed on discharge.

The OCV indicated Li_2O formation.

The gas volume measurements indicated Li_2O_2 formation.

Before Discharge

Before Wt in Air (g) =	14.82
Wt in Water (g) =	-46.22
Pressure (mm Hg) =	761
Temperature (C) =	19.8
Corrected Initial Gas Volume =	45.79
Uncorrected Initial Gas Volume =	49.04

After Discharge

Wt in Water (g) =	-37.50
Pressure (mm Hg) =	749
Temperature (C)=	21.2
Corrected Final Gas Volume =	36.87
Uncorrected Final Gas Volume =	40.32
Change in Gas Volume (Corrected) =	8.91
Change in Gas Volume (Uncorrected) =	8.72
Calculate mAh due to Lambda =	7.941 at 250 mAh/g
Total Cell Capacity =	31.93
mAh/g Due to Air =	23.989
mAh/ml =	2.69
mAh/ml (2 electron reaction)=	2.4
mAh/ml (4 electron reaction)=	4.8

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Signature

1/8/01
Date

Read and Understood Date Read and Understood Date

EXHIBIT B 7/38

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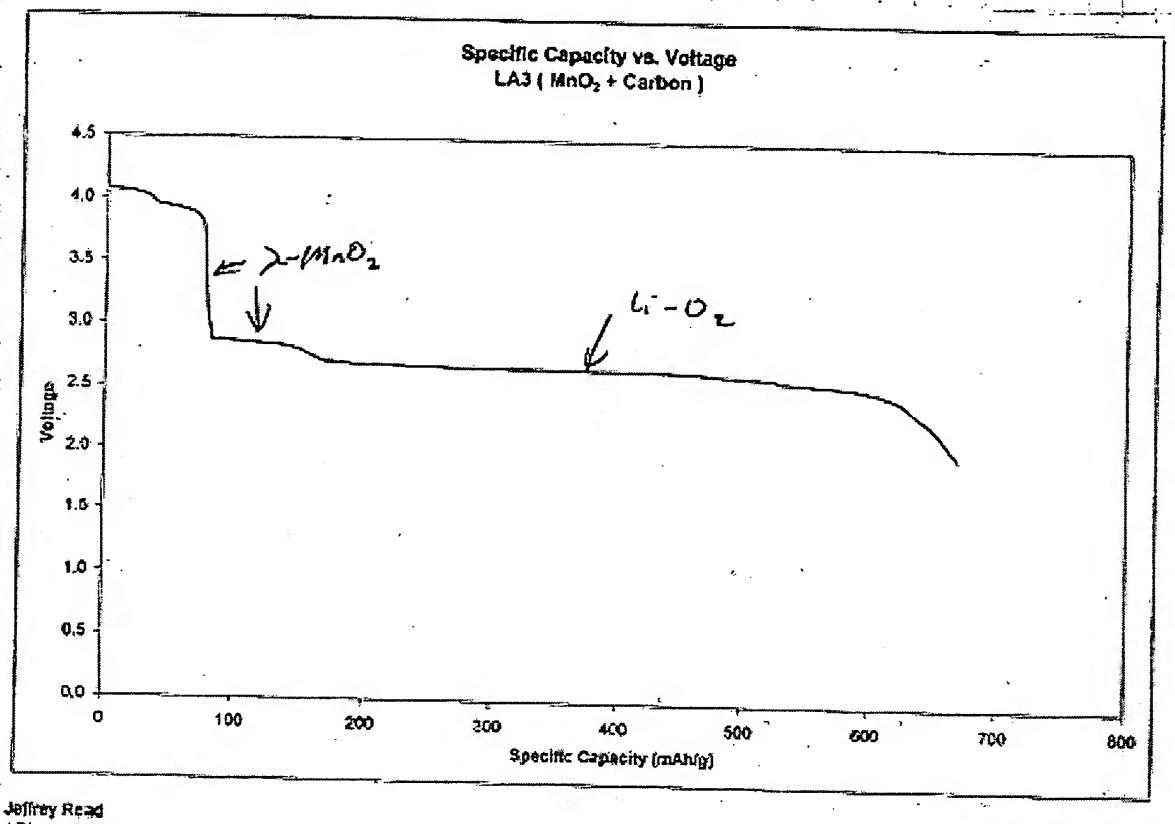
HDL № Book 8830 Page 97

9

Discharge Profile of Lithium-Air Battery LA3 1/16/01

LA3 (SC1)



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Lithium-Air Series A.xls
1/16/01

All Discharged at .1 mA to 2.0 V
Active wt. = .2187 g
Total Capacity = 146.696 mAh

The gas volume change is indicative of a 2e- reaction for oxygen reduction ie: the formation of Li_2O_2 as shown on the next page.

Continued on Page _____

Signature

1/16/01
Date

Read and Understood

Date

Read and Understood

Date

Continued from Page

Determination of 2e⁻ & 4e⁻ reaction - Li-Air 1/16/01

Before Discharge	LA1	LA3			
Before Wt in Air (g) =	14.82	15.34			
Wt in Water (g) =	-46.22	-56.81			
Pressure (mm Hg) =	761	761			
Temperature (C) =	19.8	19.8			
Corrected Initial Gas Volume =	45.79	56.16			
Uncorrected Initial Gas Volume =	49.04	60.15			
After Discharge					
Wt in Water (g) =	-37.50	-11.32			
Pressure (mm Hg) =	749	754.8			
Temperature (C)=	21.2	22.2			
Corrected Final Gas Volume =	36.87	13.47			
Uncorrected Final Gas Volume =	40.32	14.66			
Change in Gas Volume (Corrected) =	8.91	42.69			
Change in Gas Volume (Uncorrected) =	8.72	45.49			
Calculate mAh due to Lambda =	7.941	33.860	at	250	mAh/g
Total Cell Capacity =	31.93	146.696			
mAh/g Due to Air =	23.989	112.837			
mAh/ml =	2.69	2.64 ← 2e⁻			
mAh/ml (2 electron reaction)=	2.4	2.4			
mAh/ml (4 electron reaction)=	4.8	4.8			

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1/16/01
Date

Read and Understood | Date | Read and Understood | Date

Li-Air Plug Type Electrodes

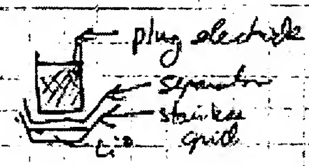
1/18/01

Electrodes were built by placing a plug of electrode material inside a 1/4" diameter pyrex tube 2.5 cm long, and placing glass rods in each end to compress the plug while being heated to 135°C.

The plug is heated for ~5 minutes until it bonds to itself.

The plug is then extracted in MeOH 3X for 20 minutes and dried under vacuum at 100°C for 2 hours.

The plug is wrapped in Aluminum grid and placed back in the glass tube and a 2 electrode cell is built vs. Li metal.



Tube dia = .235" = .597 cm
Tube X-Area = .280 cm^2

The plug electrode is held to the separator/lithium with an insulated wire and placed in a foil bag. Electrolyte is added to the cell with electrolyte being added directly to the plug electrode. The excess electrolyte is removed from the plug area and the bag is sealed with 2 leads coming out for electrical connection. The bag is cut open at one end and O_2 is put in and the bag is resealed.

The plug is put on test at .1 mA/cm^2.

Electrode material used: N3P91B   Electrolyte: N3P80B (same to LAP3; same to LAP3)

Cell #	Cath wt.	OCV	Before dis Imp. (1KHz)	Before wt in air	wt (@22°C) under (751 mmHg)	Discharge	After in Air	in ...
LAP1	.1612 g extr.	4.11	761 Ω	11.82 g	-63.90	30.0 mAh	11.82 g	-57
LAP2	.1549 g extr.	4.12	1022 Ω	12.66 g	-77.61	42 mAh	12.68 g	-86.
LAP3	.1481 g extr.	4.11	1063 Ω	10.18 g	-59.74	fully	10.21 g	-35.

LAP1 - Discharge Air portion 25% - Total Discharge = 30 mAh - Prgm - PCX1RM
LAP2 - Discharge Air portion 50% - Total Discharge = 42 mAh - Prgm - PCX1RN
LAP3 - Discharge Air portion 100% - Total Discharge = 60 mAh - Prgm - PCX1RO
*Discharge at 20.0°C

status: 3/21/01 cells LAP1 & LAP2 are discharged.
LAP3 still discharging.

4/8/01 cell finished, wts taken in air & water

Continued on Page

Signature: [signature]   Date: 3/21/01

Read and Understood ____ Date ____ Read and Understood ____ Date ____

EXHIBIT B 10/38 HDL

Continued from Page

Book 8115 Page 1

Li-Air Cells - Rate Study & Argon Blanks. 1/19/01

Cathode: N3P92A (Both 10 & 24 mils)
Electrolyte: 1 MLiPF$_6$, PC:DME (1:1) Lot: N3P90B

Cell #	Cath Thick (mil)	Cath wt (g)
LA4	10.9	.1801g
LA5	11.0	.1920
LA6	10.9	.1905
LA7	10.9	.1939
LA8	11.0	.1966
LA9	10.9	.1944
LA10	10.9	.1929
LA11	10.9	.1933
LA14	23.5	.3851
LA15	23.6	.3820
LA16	23.5	.3935
LA17	23.5	.3743
LA18	23.2	.3802
LA19	24.1	.3850
LA12	23.6	.3849
LA13	23.8	.3870

LAM's @ 300°F, 1 pass on treated Al grid.
Extracted 3X in MeOH for 30 minutes each time.

1/22/01

Dried cathodes in 100°C oven for 2 hours.

Built into 2 electrode cells w/ Lithium and Rogaine separator. Used 3-4 tissues per cathode to hold it flat.

Sealed in foil laminate -

~6 ml of 1 MLiPF$_6$ PC:DME Lot: N3P90B for each cell.

Cells for Rate Study: LA6, LA7, LA8, LA9, LA10 "Light"
LA14, LA15, LA16, LA17, LA18, "Heavy"

Cells for Argon Study: LA11, LA19

Hold backs: LA4, LA5
LA12, LA13

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Li-Air Rate Study (20°C)

Cell #	2'e (10Ω)	wt in Air	wt. in water	Discharge Rate	(After dis.) 1/31/01 wt in water	(After dis.) 2/5/01 wt. in water	1/23/01 2/8/01 weight in water
LA6	73.25Ω	15.35g	-70.80	.05 mA/cm²	still disch.	-54.01g	
LA7	67.5Ω	14.59	-35.12	.1 mA/cm²	-22.84		
LA8	71.75Ω	14.91	-81.02	.2 mA/cm²	-74.19		
LA9	65.25Ω	14.85	-62.88	.5 mA/cm²	60.57		
LA10	66.0Ω	15.88	-62.00	1.0 mA/cm²	-61.80		
LA14	69.75Ω	14.98	-90.63	.05 mA/cm²	still disch.		-59.49g
LA15	60.6Ω	15.17	-135.81	.1 mA/cm²	-114.18		
LA16	60.5Ω	14.61	-120.48	.2 mA/cm²	-107.54		
LA17	57.75Ω	15.09	-106.45	.5 mA/cm²	-103.06		
LA18	62.75Ω	14.77g	-93.79	1.0 mA/cm²	-72.56		
			21.0°C		22.3°C	22.1°C	24.0°C
			760mmHg		748mmHg	756mmHg	761.9mmHg

Li-Air Argon Blanks Study (20°C)

Cell #	2'e (Ω)	wt in Air	wt in water	Discharge rate	(After dis.) 1/31/01 wt in water	1/23/01
LA11	42.1Ω	14.53g	-15.18	.1mA/cm²	-15.61	
LA19	74.9Ω	14.98g	-16.10	.1mA/cm²	-16.31	
		21.0°C			22.3°C	
		760mmHg			744mmHg	

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EXHIBIT B 12/38

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Casting Air electrodes Vulcan XC-72 & Black Pearls

1/24/01

Cast #1

Vulcan XC-72 = 13.4603 g (30.24%)
Kynar 2801 = 6.8443 g (15.38%) > 45.62%
DBP = 24.2008 g
Acetone = 48.94 g

Dry mix, Add Acetone / DBP mix, blend for 30 seconds on setting 1
Lumpy mixture — cast @ 1.194 mm & 2.286 mm
Lot: N4P3A
Lumay FILM — LAMINATES WELL

Cast #2

Black Pearls 2000 = 13.3641 g
Kynar 2801 = 6.8830 g
DBP = 24.2427 g
Aceton = 109 g

Cast @ 2.286 mm, Thick paste, FILM CRACKED,

Li-Air Cells + Carbon Surface Area Study

1/29/01

Cell #	*Laminated Cath Th	Cell wt	Cathode Lot # N4P3A Rate	Z @ mHz	OCV	wt in Air	756.2 @ 22°C wt-in water	1/31/0
LA20	42 mils	.5810	.05 mA/cm²	34.8Ω	3.2858V	16.16g	-103.15g	-170
LA21	44 mils	.5805	.1 mA/cm²	40.9Ω	3.2877V	15.88g	-56.86g	
LA22	40 mils	.5427	.2 mA/cm²	32.8Ω	3.2924V	14.60g	-186.025	-170
LA23	43 mils	.5837	.5 mA/cm²	32.2Ω	3.2871V	15.23g	-120.30g	-113
LA24	40 mils	.5484	1.0 mA/cm²	42.5Ω	3.2904V	14.35g	-121.01g	-118

LAM1 @ 300F, 1 pass on treated Al grid
Extract 3x in meoh
dry at 100°C under vacuum for 12 hours
Seal in foil bags w/ Lithium Anode
Add ~6 drops of 1M LiPF6 PC:DME (1:1) Lot N3P908
Measure impedance after 1½ hrs
Add Pure O2
Discharge at .05, .1, .2, .5, 1.0 mA/cm²

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EXHIBIT B 13/38

Li-Air Cells

1/31/01

LA21

Li-Air cell LA21 was stopped on 1/31/01 to add more O_2.

Stopped → wt in water = -28.24g → Added O_2 → wt in Air = 14.86g → wt in water =

After Discharge: wt in water = -37.85g ← 22.9°C 725 mmHg

22.3°C 744 mm

LA22, LA23, LA24 - (were) placed back on test after they were weighed in water at .05 mA/cm² to see what the low rate capacity is.

2/1/01

Li-Air Cell (Practical)

2 - 16 cm² cathodes on Li central anode

Cathode: N4P3A

Cathode wt total = 7.45g - .19 - .23 = 7.03g

Laminated Thickness 65-75 mils

Li Anode wt = 6.3g

Separator: Rayon Type Absorber

Electrolyte Cracked when trying to build cell. Only 1/2 cell (1/2 of cath) was used.

Pinhole in package to allow O_2 entry.

Impedance ~ 10.5 Ω @ 10 Hz

Cell = LA25

Discharge at 0.2 mA/cm² = 1.6 mA

PCX1R16

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EXHIBIT B 14/38

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Li-Air Cells

2/1/01

→ Put LA8 on charge/discharge cycling for 10 cycles to see if it will recharge

Teflon Cathode Prep

2/1/01

Super P = 3.0620 g

IPA/water = 19 ml IPA to 30 ml water

17 ml of IPA/water mix added to Super P

Teflon Emulsion = 2.8378 g (61.5% Solids)

Teflon emulsion + DI water = 17.3854 g (10.03% solids soln)

Add 3 ml of Teflon/DI water mix in 1 ml aliquots. (91.05% C / 8.95% T

Mix paste after each aliquott

Paste onto Treated Al grids

Put on blotting paper w/ weight then copy paper + wt

Allow to dry with weight overnight.

2/2/01

→ Cathodes cracked badly

→ Pressed on Carver at 4000 & 9000 pounds for 16 cm² size coating.

→ built into Li-Air cells

Cell #	cath grid wt	pressed at	Z'e Ωhr	grid ~~w/ tab~~
LA26	.0886 g	9000 lb	10.5 Ω	
LA27	.1182 g	9000 lb	17.7 Ω	
LA28	.1685 g	4000 lb	19.7 Ω	
LA29	.1612 g	4000 lb	31.0 Ω	

Activated w/ 6 ml of 1M LiPF₆ PC:DME Lot N3P90B

Run Impedance

Filled w/ O_2

Tested at .5 mA/cm²

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Signature [signature] Date 2/2/01

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Cells w. Teflon cathodes - Additional Testing 2/5/01

Cells LA27, 28 & 29 were put back on test to discharge at .5mA/cm² after being recharged to various potentials at .2mA/cm².

Cell	Charge to	Test	Filename
LA27	3.20V	RCX5-A	LA27B
LA28	3.50V	RCX5-B	LA27C
LA29	4.15V	RCX5-C	LA27D

Cell LA26 was destroyed.

Preparation of 10% solid Teflon solution 2/6/01

Teflon 30 Emulsion (61.5% solids) = 36.8713 g

DI water = ~~197.91g~~ 191.91g

~~36.8713 x .615 x 100 / (197.91 + 36.87)~~

$\frac{36.8713 \times .615 \times 100}{(191.91 + 36.87)}$ = 9.91% Teflon

Preparation of Teflon paste for cathodes 2/6/01

IPA/water = 13.7ml IPA diluted to 39ml with DI water

Super P = 3.0986g

20ml of IPA/water added to Super P + 7ml Teflon 30 (10% Emlsn - 9.91% actual) = 1.06g + 1.08g + 1.06g

Mix to shiny paste. over →

EXHIBIT B 16/38

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Teflonated electrodes

2/6/01

8/31 Grid	Grid wt.
1	.0375
2	.0403
3	.0408
4	.0401
5	.0399
6	.0390

The Sipa P/Teflon 30 paste from #6 was coated onto the Aluminum grids 1-6 Above. The paste was put on both sides of the grid and then a 23 mil shim was used to make a level, constant thickness electrode. The excess paste was squeezed out. The electrodes were between Al foil during the leveling process and then left there to dry under a constant weight at 85°C in a constant temperature oven.

When dried the electrodes cracked and upon pressing/testing they did not form good electrodes.

Impedance measurements on discharged cells

2/13/01

Cell	After Discharge: Imp. @ 10 Hz	After Discharge: Imp @ 3x10⁴ Hz	Before Discharge: Imp @ 10 Hz	Before Discharge: Imp @ 3x10⁴ Hz
LA8	35.0 Ω	.090 Ω	71.7 Ω	.100 Ω
LA1	31.6 Ω	.102 Ω	67.0 Ω	.110 Ω
LA3	32.5 Ω	.095 Ω	53.8 Ω	.111 Ω
LA6	45.1 Ω	.091 Ω	73.2 Ω	.096 Ω
LA7	49.9 Ω	.095 Ω	67.5 Ω	.099 Ω
LA9	57.6 Ω	.095 Ω	65.7 Ω	.101 Ω
LA10	58.3 Ω	.096 Ω	66.0 Ω	.097 Ω
LA11	34.6 Ω	.099 Ω	42.1 Ω	.102 Ω
LA14	46.5 Ω	.094 Ω	69.2 Ω	.103 Ω
LA15	43.4 Ω	.093 Ω	60.6 Ω	.101 Ω
LA16	55.9 Ω	.093 Ω	60.5 Ω	.101 Ω
LA17	56.7 Ω	.069 Ω ???	52.7 Ω	.106 Ω
LA18	49.1 Ω	.094 Ω	62.7 Ω	.101 Ω
LA19	67.3 Ω	.092 Ω	74.9 Ω	.103 Ω
LA21	27.8 Ω	.093 Ω	40.9 Ω	.097 Ω
LA27	44.0 Ω	.096 Ω	17.6 Ω	.096 Ω
LA28	22.7 Ω	.100 Ω	19.7 Ω	.096 Ω
LA29	8.7 Ω	.094 Ω	31.0 Ω	.095 Ω

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EXHIBIT B 17/38

Cut-off Voltage Studies of Li-O$_2$ cells 2/15/01

Cells LA 4, 5, 12 & 13 (pvl) were activated by adding ~6ml of 1 Mull. PC:DME (1:1) Lot # N3P90B.

Cells will be charged and discharged between 2 different voltag

LA 4, LA 12 - 4.15V to 2.0V
LA 5, LA 13 - 4.15V to 2.5V

Cell	Impedance	OCV	Test	Channel	Test
LA4	62.5Ω	4.118	4.15V→2.0V	1	LA×1B
LA5	53.3Ω	4.114	4.15V→2.5V	32, Aux 1	LA×1C
LA12	52.5Ω	4.117	4.15V→2.0V	3	LA×1B
LA13	53.6Ω	4.119	4.15V→2.5V	5	LA×1A

LA5 - wt in Air before cycling = 13.87g

LA5 - wt in water before cycling = 160.95g (752.5 mm Hg, 23.1°C)

2/20/01

Cells LA27, LA28 and LA29 were put back on discharge at 1. after the impedance testing on 2/13/01.

Cell	Test	Filename
LA27	PCX1A	LA27C
LA28	PCX1A	LA28C
LA29	PCX1A	LA29C

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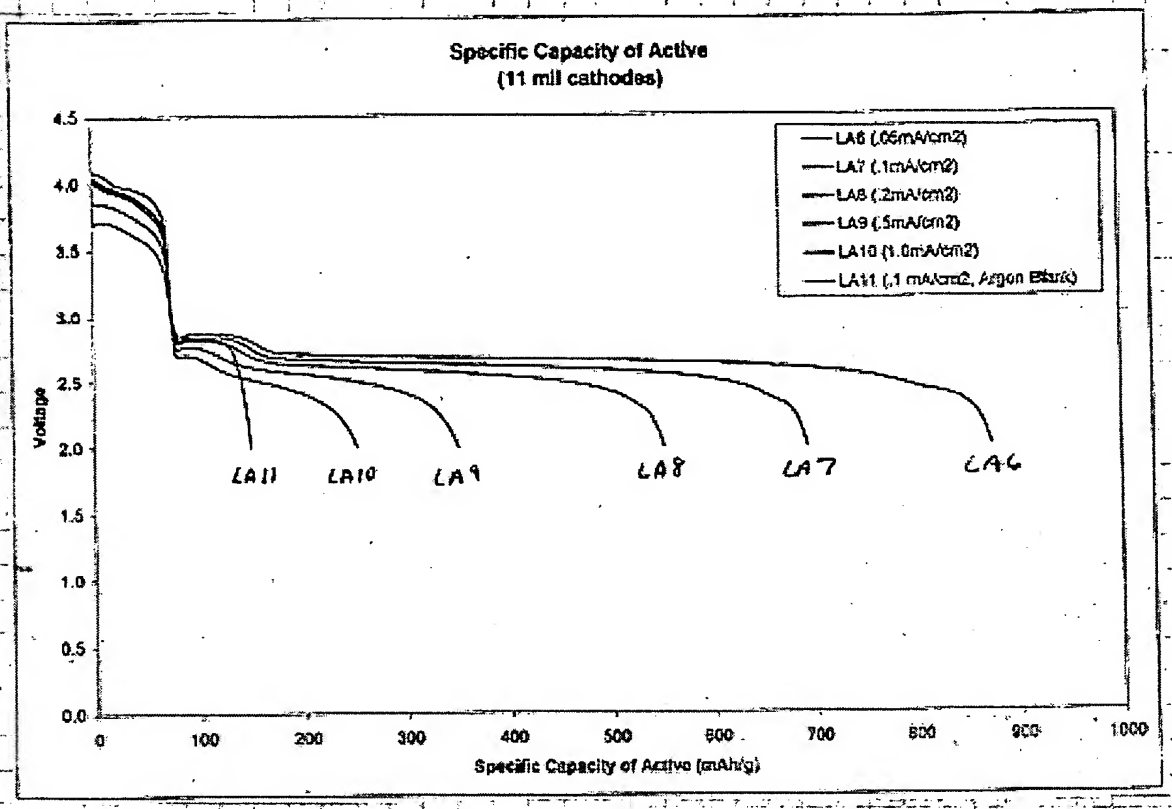
EXHIBIT B 18/38

Results for Li-Air Cells LA6 - LA11 3/21/01



The Specific Capacity of the Active components (λ-MnO_2 + Carbon) decreases quite rapidly with discharge rate. The specific capacity of the electrode in the absence of O_2 is shown in LA11 where only the λ-MnO_2 capacity discharges.
The limited rate capability may be due to slow kinetics of the O_2 reduction or O_2 solubility in the electrolyte.

The energy density plot for these cells shows a maximum of nearly 2500 Wh/kg at .05 mA/cm², extremely high for any battery system.

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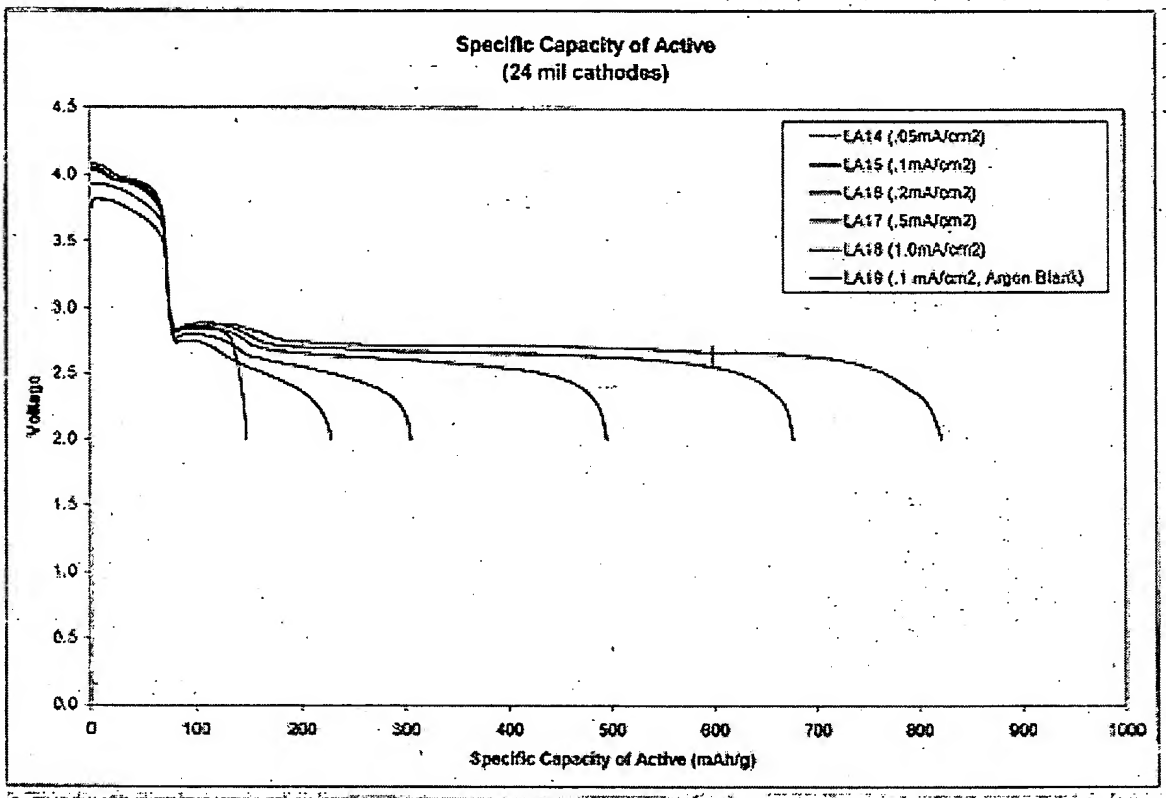
Results for Li-Air Cells LA14-LA19 3/21/01



Cells LA14-LA19 show identical behavior to those of LA16-LA11 with some slight decrease in capacity due to the thickness of the electrodes being 24 mils instead of 12 mils

	Spec Cap Active (mAh/g)		Spec Cap Carbon (mAh/g)		Specific Cap of Cathode (mAh/g)		
rate	11 mils	24 mils	11 mils	24 mils	11 mils	24 mils	Lambda
0.05	874	822	1500	1769	579	545	
0.1	693	677	1429	1388	459	449	183
0.2	552	496	1059	912	366	328	
0.5	351	305	531	411	232	202	161
1.0	253	229	273	210	167	151	155

The table summarizes the results for: Specific Capacity of Active (λ-MnO_2+C), Specific Capacity of Carbon (C), Specific Capacity of Cathode (λ-MnO_2+C)

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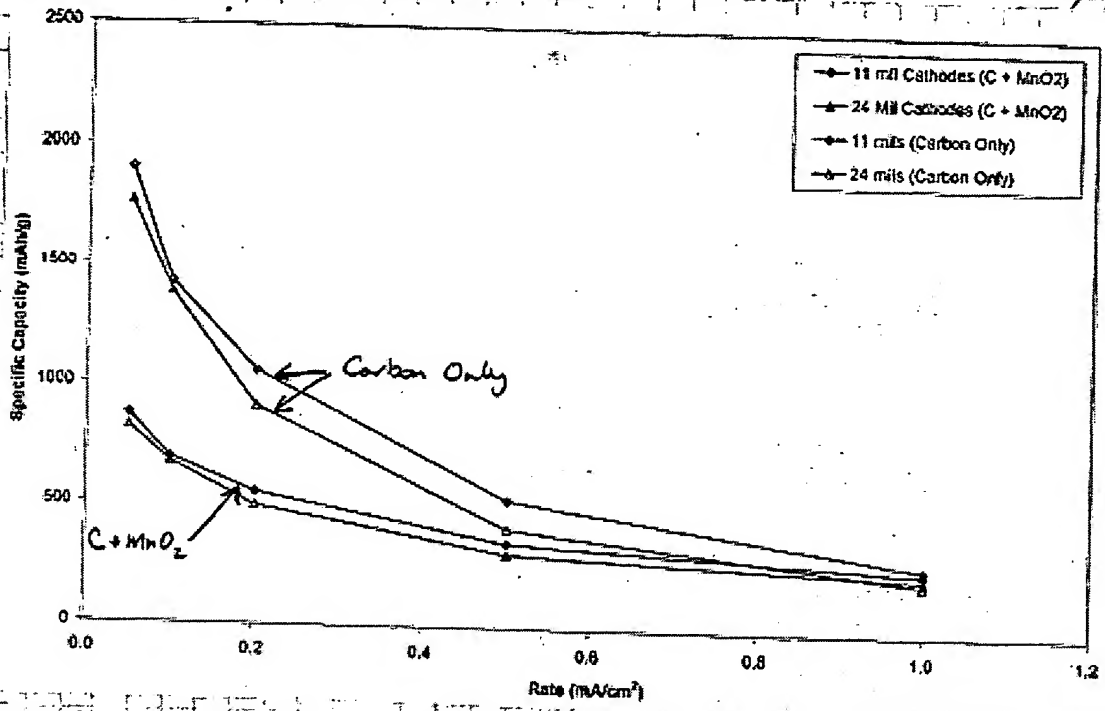
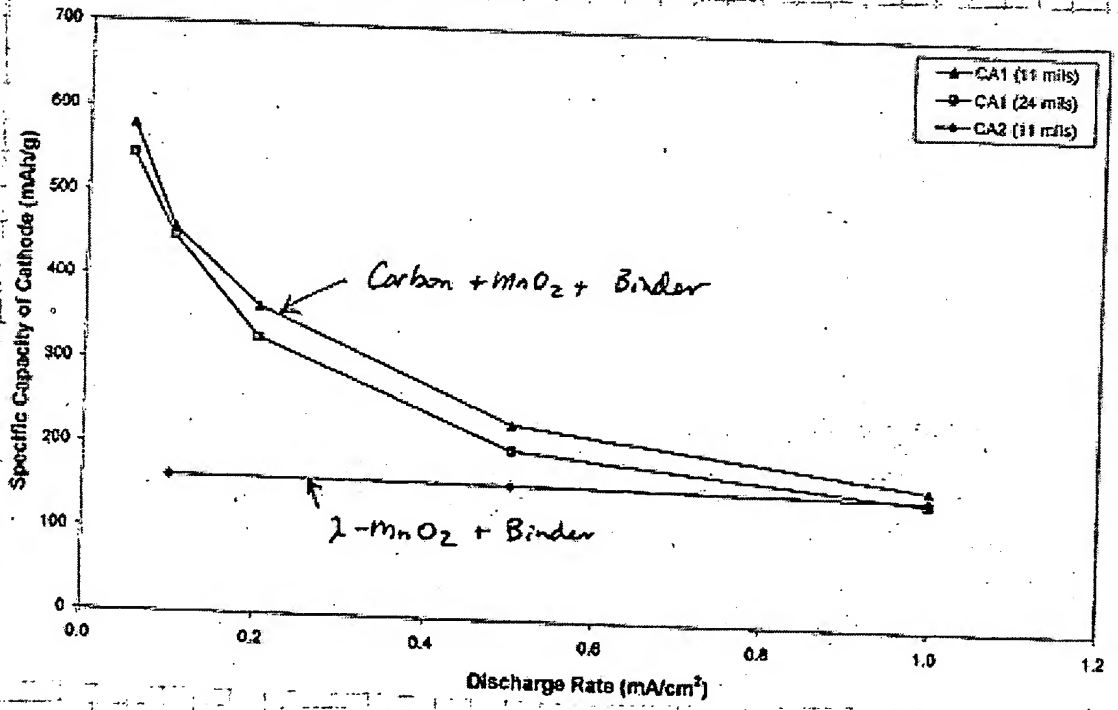
Rate Curves for LA16 - LA19 3/21/01





The Specific Capacity of the working Cathode is much higher in the Li-Air cell than in the Li-λ-MnO2 cell

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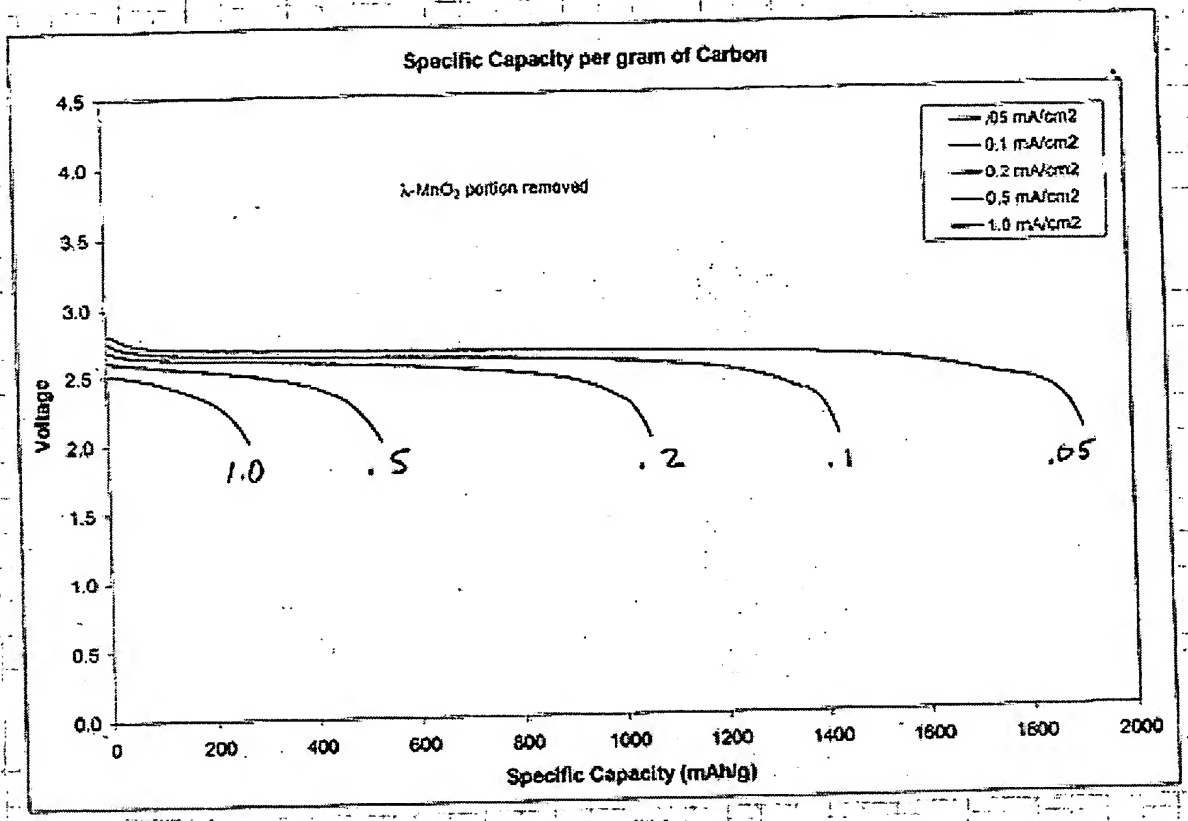
Li-Air Cells LA6-LA10 (Carbon Specific Capacity) 3/21/01



With the specific capacity of the carbon only is calculated based on the Super P capacity, the total specific capacity is exceedingly high. If this type of energy can be realized in a D-size cell with 90% carbon, A extremely high energy density ce can be realized.

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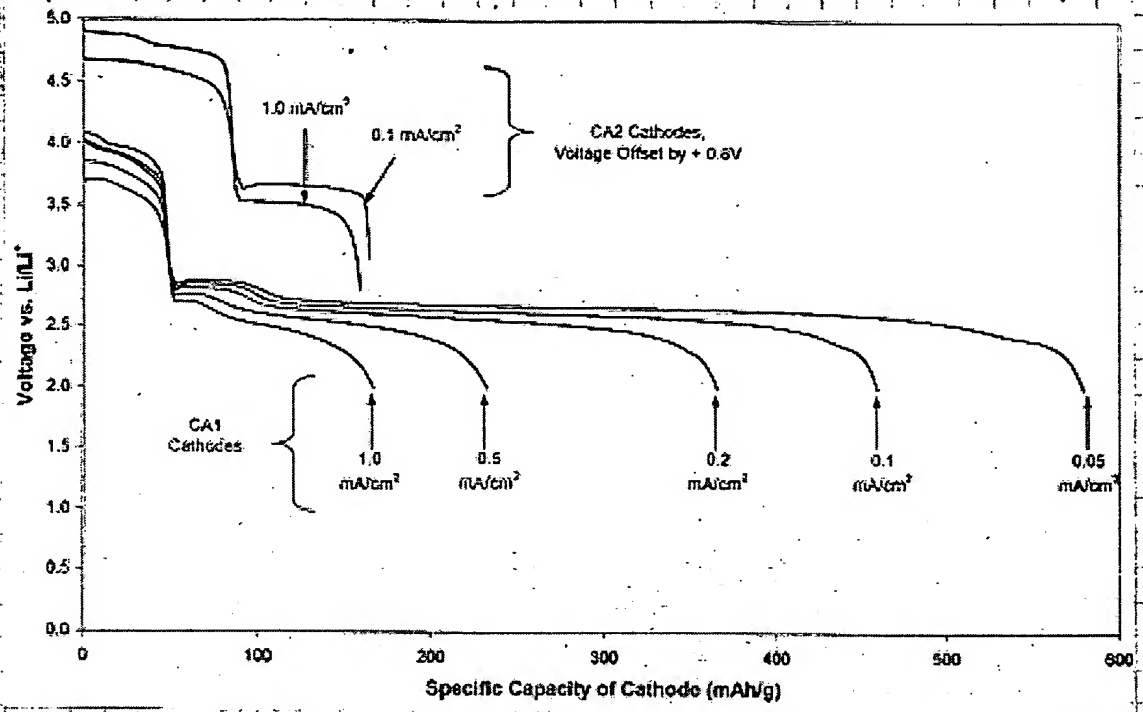
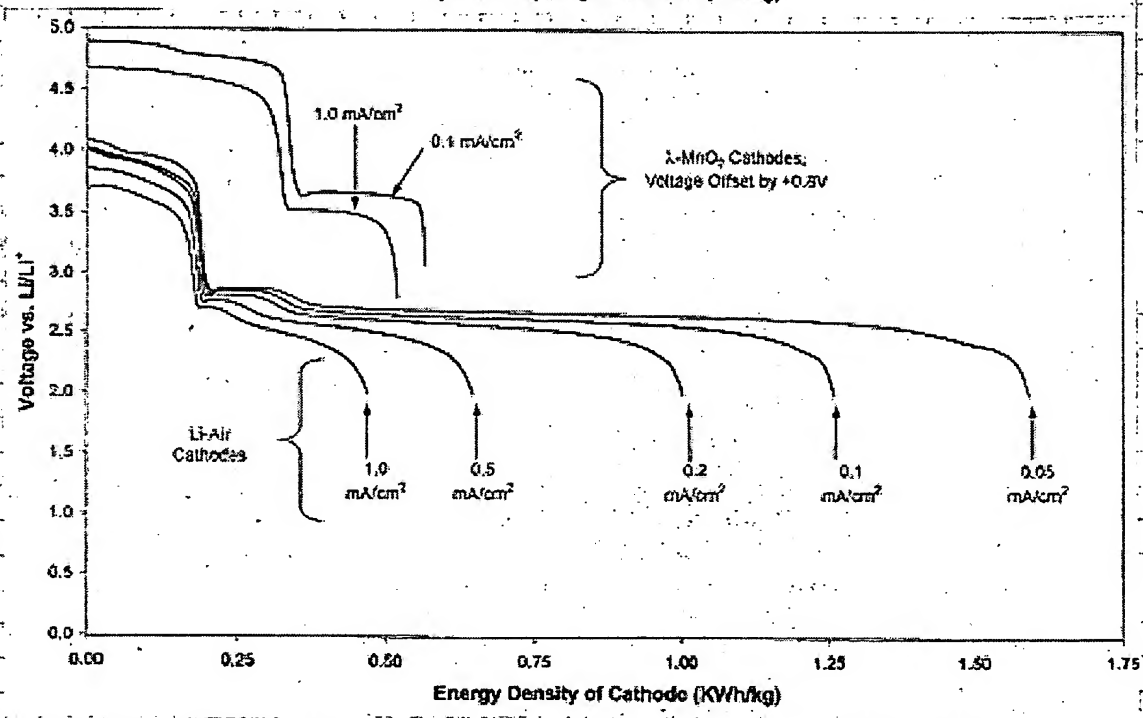
Comparison of Li-Air to λ-MnO_2 3/21/01





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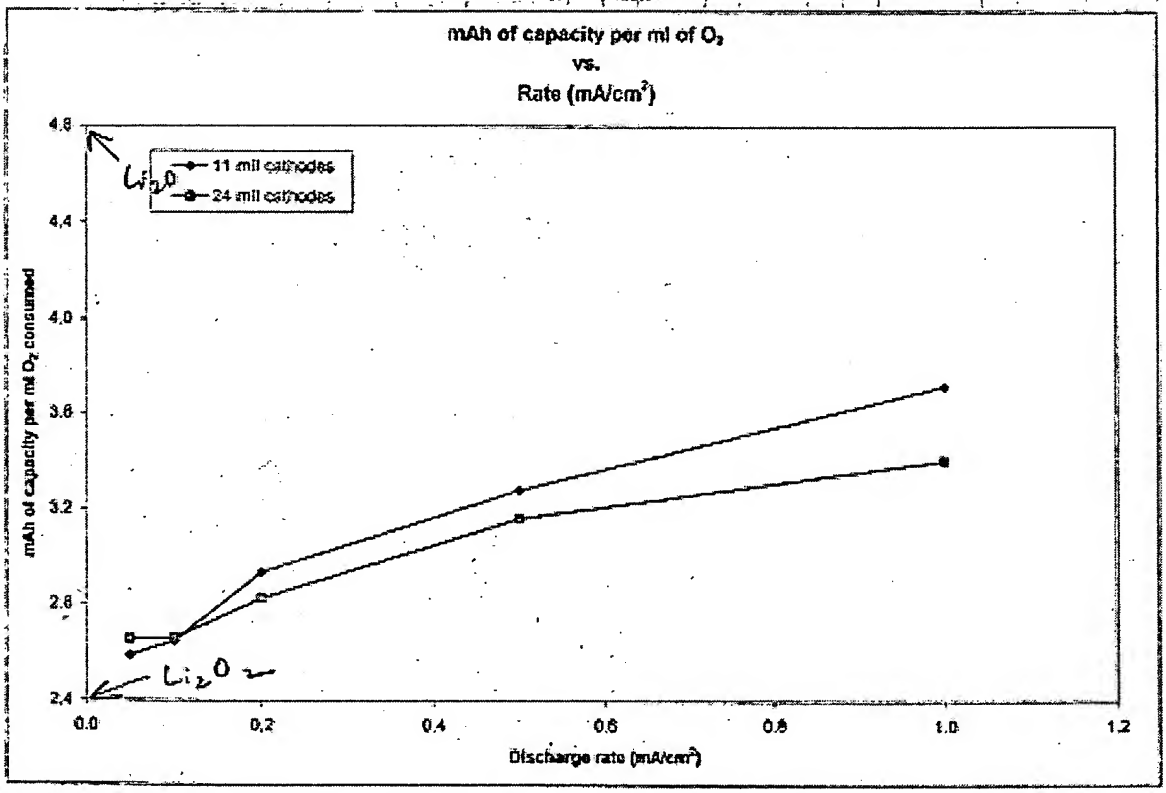
O_2 gas consumption on Discharge of Li-Air Cells 3/21/01

Gas consumption (pure O_2) was measured for LA6-LA19 after discharge at various rates. The gas consumption/mAh decreased as discharge rate increased. The mAh/ml O_2 for the formation of Li_2O_2 is theoretically 2.4 mAh/ml and for Li_2O 4.8 mAh/ml. The graph shows that at low rates Li_2O_2 is formed and as rate increases the formation of Li_2O becomes more pronounced.



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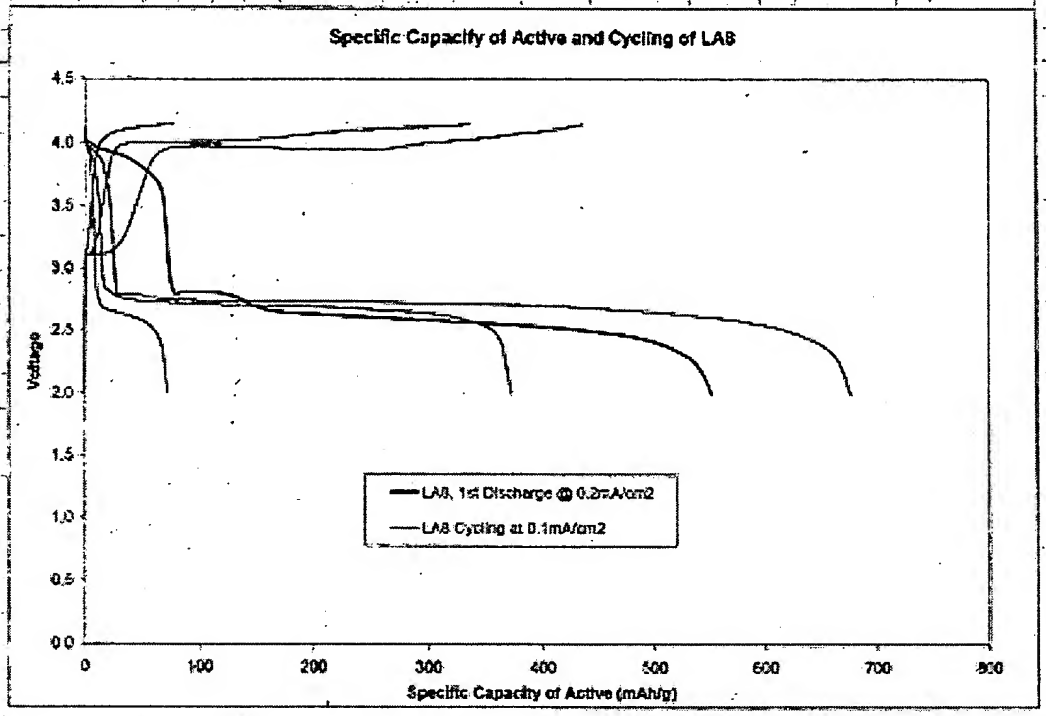
LA8 Cycling -0.2mA/cm² initial discharge, .1mA/cm² cycling 3/21/01

LA8 was initially discharged at 0.2mA/cm² for rate studies. The cell was taken off of test then put back on to cycle at 0.1mA/cm² between 4.15V and 2.0V.



The 1st discharge at 0.2 mA/cm² was 550mAh/g Active. Upon charge, 436 mAh/g was recovered. The 2nd discharge was 676 mAh/g. The third, 373, The fourth, ~~[illegible]~~ 73mAh/g.

The capacity fades quickly as the ability of the cell to charge decreases rapidly. The capacity appears to be limited by the cells ability to recharge.

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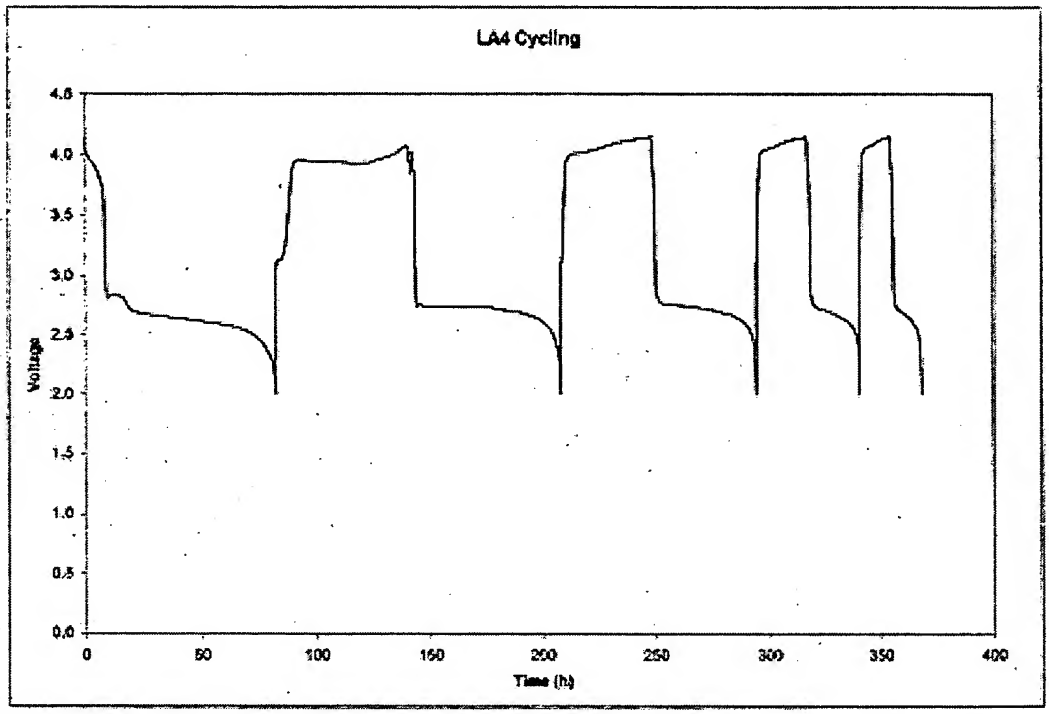
LA4 cycling 3/21/01.

There were several cells built simply designed to cycle at low rate. LA4 was such a cell cycled between 4.15 V and 2.0 V at 0.1 mA/cm^2. LA5 was cycled between 4.15 V and 2.5 V at 0.1 mA/cm^2.

The cycling of LA4 is shown below. The capacity fade is apparent on successive cycles.



LA5 showed nearly identical behavior to LA4 even with the higher (2.5 V) cutoff.

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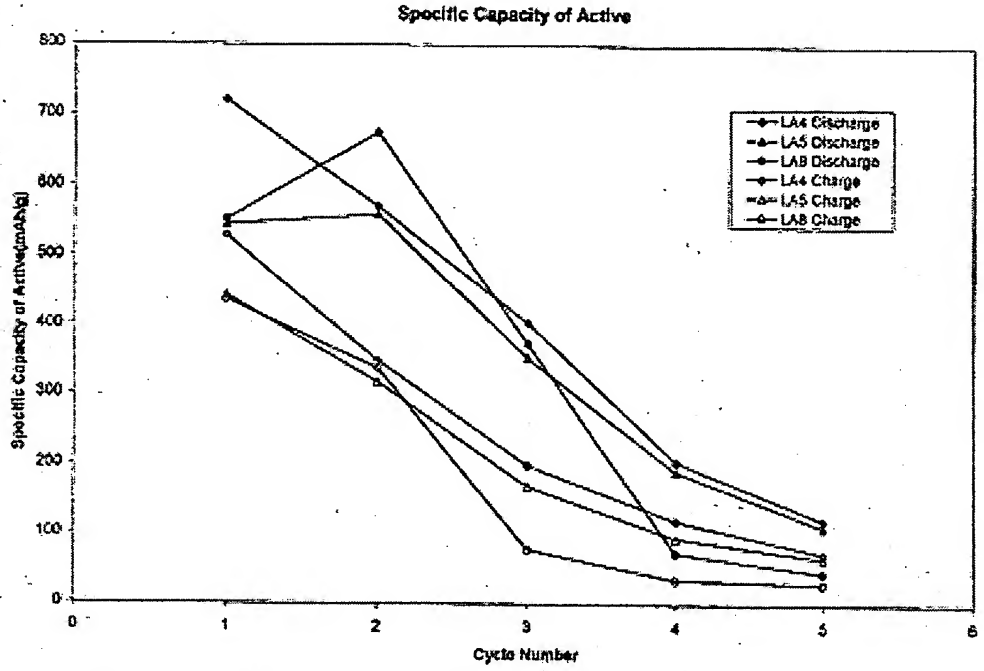
LA4, LA5 and LA8 Cycling. 3/21/01

The cells LA4-8 all cycled in similar fashion with the charge capacity determining capacity fade.



Specific Capacity of Active Components

Cycle	LA4		LA5		LA8	
	Discharge	Charge	Discharge	Charge	Discharge	Charge
1	721	530	545	442	552	436
2	571	348	559	317	676	337
3	403	199	352	169	373	78
4	205	120	191	95	73	35
5	121	72	111	66	45	29
	2021	1269	1758	1089	1719	915

The specific capacity of the active components is extremely high, but holds only for several cycles.

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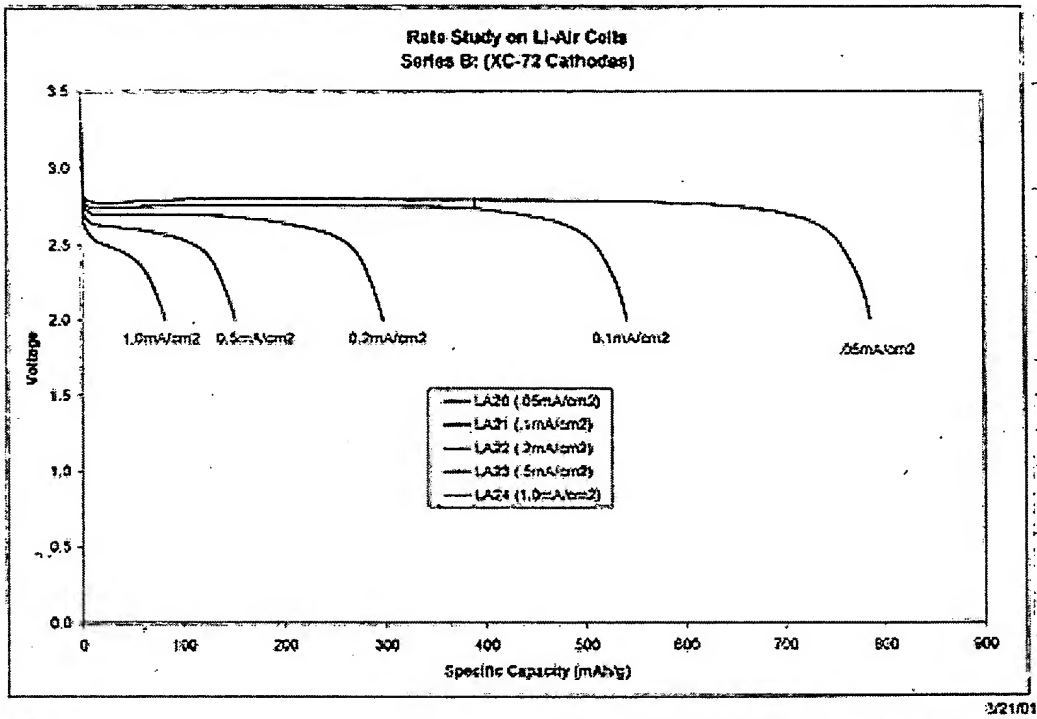
Li-Air Cells LA20-24 : (XC-72 Carbon.) 3/22/01



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(Series B)

Cells built with higher surface area carbon demonstrated similar performance to the Super P carbon used in cells LA4-LA19. Based on carbon weight alone, the higher surface area material actually performed at a lower capacity.

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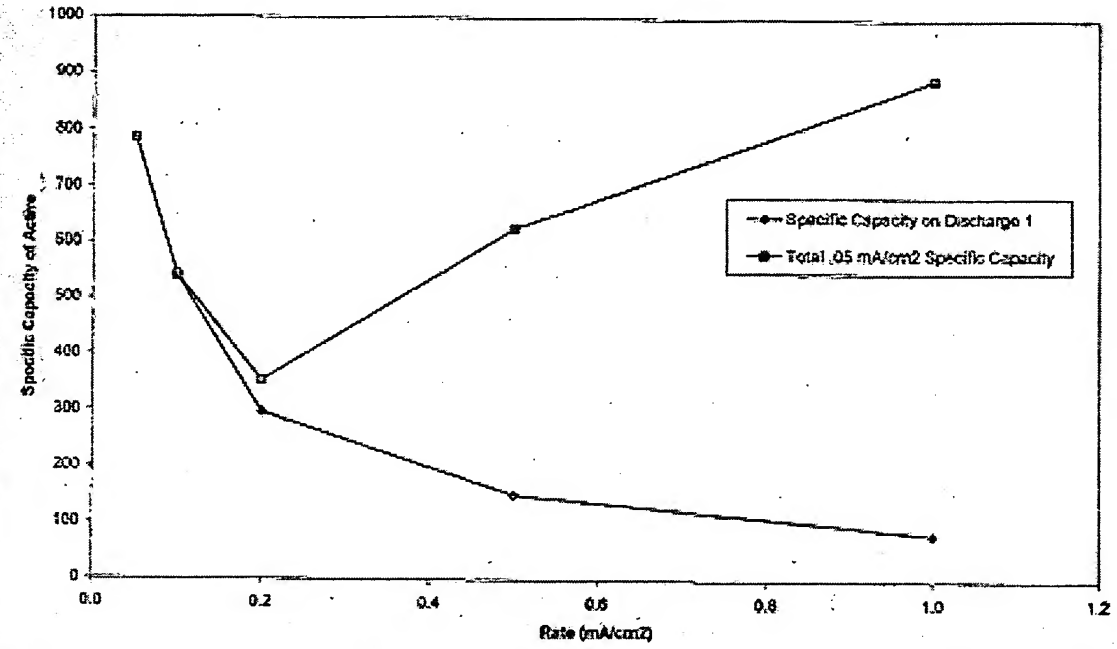
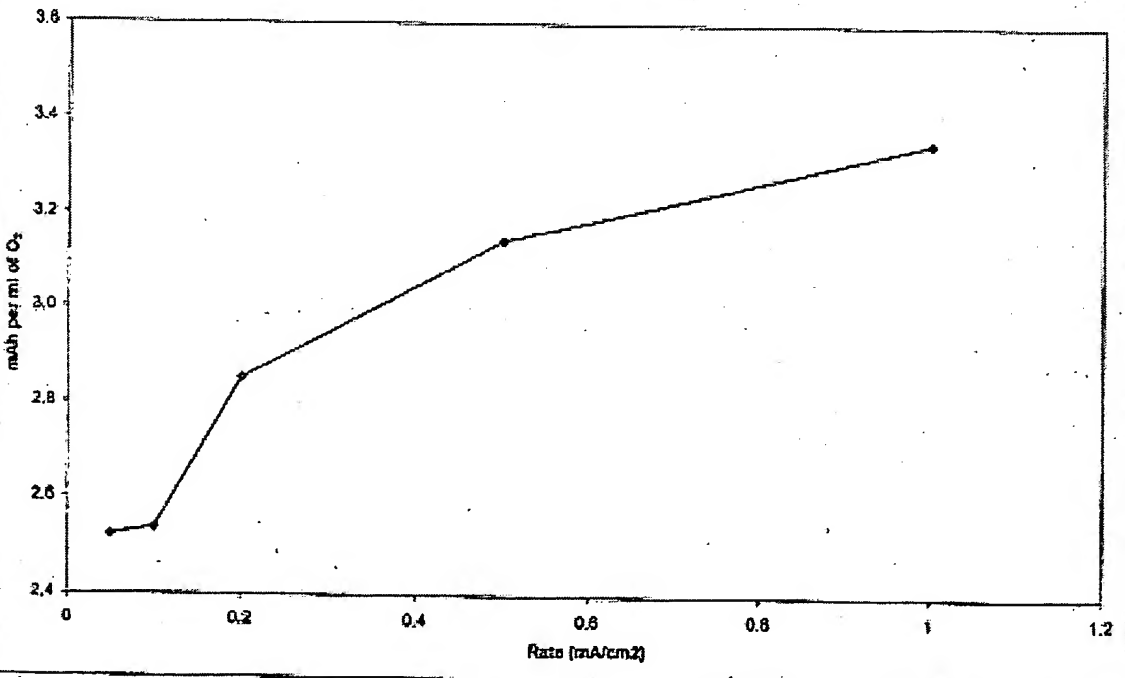
Li-Air Cells LA20-LA24 (XC-72 Carbon) 3/22/01

Specific Capacity vs. Rate for Discharge 1
and
Total Specific Capacity with Discharge 2 at .05mA/cm²



SERIES B
mAh of Capacity per ml O_2 v. Discharge rate (mA/cm²)



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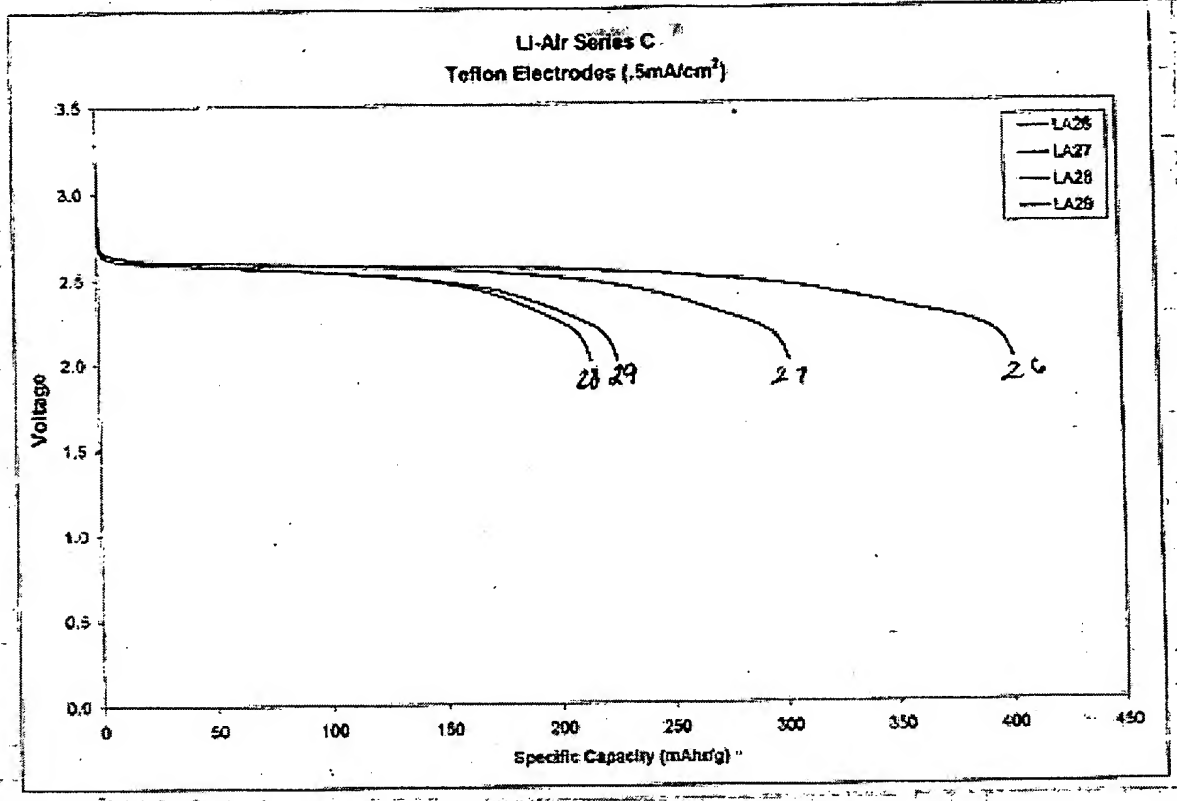
Series C

Li-Air Cells LA26-LA29 Teflonated Electrodes, Super P 3/2



- Teflonated Electrodes of Super P (91% carbon) were discharged at .5mA/cm².
- The electrodes performed similarly to the Super P in LA11-LA19. That on a per gram carbon basis, the capacity was similar.
- Upon discharge of cells LA27, 28 & 29 at .1mA/cm², an additional (at 2V) 500, 315 & 485 mAh/g of capacity is extracted.

Jeffrey Ahrd 3/22

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EXHIBIT B 30/38

HDL

MICA Grid Treatment Prep. (1:1 MICA : SuperP) 3/28/01

Super P = 2.7014 g

IPA = 54.9690 g

DI Water = 54.0280 g

MICA D-709 = 13.0050 g

The mixture was homogenized for 10 minutes on level 4 on the PRO 250

1M $LiPF_6$ - γ-BL Electrolyte Prep. (50 ml) 3/28/01

γ-BL Water Runs: Run 1: 33.2 µg/2 ml = 16.6 µg/ml

Run 2: 32.2 µg/2 ml = 16.1 µg/ml

$LiPF_6$ = 7.8718 g

1M $LiPF_6$ - γ-BL final volume ≈ 46 ml

Lot#: N4P23A

Electrolyte turned brown after several days 3/30/01

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[signature] 3/30/01

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EXHIBIT B 31/38

Electrolyte Studies for Li-Air (Series A) 3/30/01

Cathode: N3P9ZA (10 mils)
Electrolytes: 1M LiPF$_6$ w/ PC
1M LiPF$_6$ w/ γ-Butyrolactone
1M LiPF$_6$ w/ EC/DMC (2:1)

Cell #	Cathode wt.	Separator	Electrolyte (~3mL) 4/2/01	Test
LA30	.1881	Celgard 2300	1M LiPF$_6$ PC:DME (1:1) N4P9A	Gas Volum
LA31	.1874		1M LiPF$_6$ EC:DMC (2:1) N3P9ZA	Cycling
LA32	.1930		1M LiPF$_6$ EC:DMC (2:1) N3P82A	Cycling
LA33	.1880		1M LiPF$_6$ PC:DME (1:1) N4P10A	Gas Volu
LA34	.1910		1M LiPF$_6$ PC:DME (1:1) N4P10A	Gas Volu
LA35	.1879		1M LiPF$_6$ PC:DME (1:1) N4P10A	Gas Volu
LA36	.1843		1M LiPF$_6$ PC:DME (1:1) N4P17A	Gas Volu
LA37	.1893	Rayovac	1M LiPF$_6$ EC:DMC (2:1) N3P82A	Rate Study
LA38	.1884		1M LiPF$_6$ EC:DMC (2:1) N3P82A	"
LA39	.1836		1M LiPF$_6$ EC:DMC (2:1) N3P82A	"
LA40	.1864		1M LiPF$_6$ PC N4P25A	"
LA41	.1861		1M LiPF$_6$ PC N4P25A	"
LA42	.1839		1M LiPF$_6$ PC N4P25A	"
LA43	.1885		1M LiPF$_6$ γ-BL N4P23A	"
LA44	.1842		1M LiPF$_6$ γ-BL N4P23A	"
LA45	.1802		1M LiPF$_6$ γ-BL N4P23A	"
LA46	.1861		1M LiPF$_6$ PC:DME (1:1) N4P10A	40°C
LA47	.1846		1M LiPF$_6$ PC:DME + Trimoxy N4P17A	Rate
LA48	.1782		1M LiPF$_6$ PC:DME + Trimoxy N4P17A	"
LA49	.1845		1M LiPF$_6$ PC:DME + Trimoxy N4P27A	"
LA50	.1754		1M LiPF$_6$ PC:DME N4P33C	Rate
LA51	.1808	Rayovac	1M LiPF$_6$ PC:DME N4P33C	Rate
LA52	.1788		1M LiPF$_6$ PC:DME N4P33C	Rate
LA53	.1698		1M LiPF$_6$ PC:THF N4P33A	Rate
LA54	.1790		1M LiPF$_6$ PC:THF N4P33A	"
LA55	.1751		1M LiPF$_6$ PC:THF N4P33A	"
LA56	.1699		1M LiPF$_6$ PC:THP N4P33B	"
LA57	.1736		1M LiPF$_6$ PC:THP N4P33B	"
LA58	.1706		1M LiPF$_6$ PC:THP N4P33B	"
LA59	.1739			

Built into cells on 4/2/01

Built into cells on 4/11/01

LAm 1 @ 300°F, 2 passes
Aluminum grid treated w/ 1:1 mICA:Superp

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1M $LiPF_6$ PC Electrolyte Prep. 4/3/01

PC Water Test: 2 ml @ 37.6 µg = 19 µg/ml

2 ml @ 39.0 µg = 19 µg/ml

50 ml electrolyte prep

$LiPF_6$ = 7.8752 g

Lot # N4P25A

Cycling Study on Li-Air cells CA31, CA32 4/5/01

1. Heat for 3 hours
2. Discharge @ 1mA/cm² to 2.0V
3. Charge and Discharge between 4.15V and 2.0V @ 1mA/cm²
4. Cycle 15 cycles.

Test Name: CAX1B.

Cell	Before wt. in Air	Before wt. in water 761, 20C	Ch.	After wt. in Air	After wt. in water 750, 22°C
LA31	13.88 g	-63.89 g	1	13.90 g	-53.77
LA32	13.75 g	-86.15 g	2	13.76 g	-76.97

Gas Volume Study # 1 4/5/01

Cell	wt. in Air	Before wt. in water 761, 20°C	After Discharge wt. in water 751, 21°C	wt. in water
LA30	12.35 g	-113.27	-102.64	12.40 g

Ran all on PCX1R. (1mA/cm²).

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Rate Study on Li-Air w/ 3 electrolytes 4/9/01

(Series A)

Run impedances on LA37 - LA45

LA37 - LA39 → 150 - 220Ω (1MLiPF6 EC:DMC (2:1))

LA40 - LA42 → 1000 - 1500Ω (1MLiPF6 PC) Poor Sep. wetting?

LA43 - LA45 → 40 - 60Ω (1MLiPF6 γ-BL)

Cell	wt in air	Before Disch. wt in water 751, 21°C	Ch.	Rate	wt in air	After Disch. wt in water 750, 22°C
LA37	13.17g	-59.04	3	.05mA/cm²	13.17g	-48.70
LA38	12.94g	-69.16	4	.2mA/cm²	12.91g	-65.72
LA39	13.00	-59.52	5	1.0mA/cm²	13.00g	-59.20
LA40	13.35	-59.24	6	.05mA/cm²	13.35g	-54.94
LA41	13.18	-64.40	7	.2mA/cm²	13.18g	-62.86
LA42	13.35	-48.37	8	1.0mA/cm²	13.35g	-48.57 -48.
LA43	12.95	-61.08	20	.05mA/cm²	12.93g	-51.51
LA44	13.31	-68.67	21	.2mA/cm²	13.31g	-64.88
LA45	13.31	-68.43	22	1.0mA/cm²	13.31g	-68.03

All cells were discharged after a 3h rest to 2.0V at the rate specified

4/12/01

Rate Study on Li-Air w/ Triton 100x added PC:DMC Elect.

(Series A')

4/10/01

Run Impedances:

LA47, LA48, LA49 → 30-40Ω

After Disch (4/13/01)

Cell	wt in air	Before Disch: wt in water	Ch.	Rate	wt in air	wt in water
		21.5°C 954mmHg				25°C/759mmHg
LA47	11.85	-59.01	46	.05mA/cm²	11.87g	-40.17g
LA48	12.60	-66.20	47	.2mA/cm²	12.62g	-57.12g
LA49	12.75	-84.53	48	1.0mA/cm²	12.75g	-83.07g

Cells discharged at given rate to 2.0V after 3h rest.

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Heat Treatment of AB-Grade EMD (-325 mesh) 4/11/01

Lot #: N3P83A (Initial Sieved Powder)

Initial wt = 127.1472 g

Final wt = 121.0326 g

380°C for 24 hrs (1:05p 4/11/01 - 12:30 4/12/01)

Lot # N4P27B

4/12/01

Prep of 1% Triton-100X PC:DME Electrolyte

Triton 100X = 1100 g

Dissolved into 1000 ml of 1M $LiPF_6$ PC:DME (N4P10A)

Lot # N4P27A

4/12/01

GAS Volume Study #2

Cell	wt in Air	Before disch. wt in water	After disch wt in water	After disch. wt in Air
GA33	12.89 g	-62.74 g, 21.5°C, 759	-59.75, 21.2°C, 751	12.91 g

Ran on PCX5R (.5 mA/cm²)

Initial Buret Volume = 23.8 cc before equilibration
28 cc Near equil.

Final Buret Volume = 32 cc

Impedance - 38 Ω @ 10 Hz.

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HT of Ken-McGee AB Grade EMD 4/12/01

Lot# 1659 (Not Sieved)

Initial wt = 82.1530g Final wt = 78.5832

HTC 380°C in air for 24 hours (2:30p 4/12/01 - 1:35p 4/13/01)

Lot# N4P28A

4/13/01

Gas Volume Study #3

cell	Before wt in air	wt in water 24.5°C, 751mHg	After wt in air	wt in water 22.0°C, 750	Impedance
LA34	12.38g	-89.76g	12.40g	-79.86	485Ω @ 10Hz

Run on PCR05R PCX2R (.2mA/cm²)

4/16/01

HT of Ken-McGee AB Grade EMD (+325 mesh)

Lot# N3P83B (+325)

Initial wt = 100.7283g Final wt = 95.9547

HT@ 380°C in air for 24hrs (noon 4/16/01 - 6pm 4/17/01)

LOT# N4P28B

4/16/01

HT of λ-MnO2

Lot# AW-46

Initial wt = 11.7872g Final wt = 11.6864 4/18/01

HT @ 380°C in air for 24 hours (noon 4/16/01 - 6pm 4/17/01)

Lot# N4P28C

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EXHIBIT B 36/38 HDL

Water Determinations on dried Solvents 4/18/01

PC:DME (N4P31A)

Run 1: 1.1 ml @ 31.5 µg = 28.6 µg/ml To High
Run 2: 2.0 ml @ 56.3 µg = 28.2 µg/ml

δ-BL

Run 1: 1.0 ml @ 201.7 µg = 201.7 µg/ml To High
Run 2: 1.0 ml @ 178.0 µg = 178 µg/ml

THP

Run 1: 1.0 ml @ 20.8 µg = 20.8 µg/ml
Run 2: 2.0 ml @ 26.1 µg = 13.0 µg/ml OK to USE
Run 3: 0.4 ml @ 6.8 µg = 17.0 µg/ml

THP

Run 1: 1.1 ml @ 14.9 µg = 13.5 µg/ml OK to USE
Run 2 2.0 ml @ 26.2 µg = 13.1 µg/ml

1,4-Dioxane

Run 1: 1.1 ml @ 16.5 µg = 15.0 µg/ml OK to USE
Run 2: 2.0 ml @ 29.0 µg = 14.5 µg/ml

4/23/01

PC:DME (N4P31A)

Run 1: 2.0 ml @ 30.3 µg = 15.2 µg/ml OK to USE
Run 2: 2.0 ml @ 29.9 µg = 15.0 µg/ml

δ-BL

Run 1: 1.0 ml @ 55.6 µg = ~~27.8~~ µg/ml 55.6 µg/ml
Run 2: .5 ml @ 31.7 µg = 63.4 µg/ml
Run 3 1.0 ml @ 54.9 µg = 54.9 µg/ml

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Preparation of ~~Cyclic~~ Cyclic Ethers for PC:Ether JR

4/23/01

Preparation of Electrolytes for Rate Study PC: Cyclic Ethers

Electrolytes to Prepare: PC:THF, PC:THP, PC:1,4 Dioxane all 1:1 with $LiPF_6$: FW = 151.9 g/mole

50 ml each prep.

1M $LiPF_6$
PC:THF (1:1)

PC: 13.939 g + 5.148 g + 4.620 + .440 g = 24.147 g
THF: 15.399 g + 3.458 g + 4.045 g + 1.027 g = 23.929 g
$LiPF_6$: 7.559 g

Lot# N4P33A

1M $LiPF_6$
PC:THP (1:1)

PC: 15.347 g + 8.139 g + .490 g = 23.976 g
THP: 16.501 g + 7.092 g + .421 g = 24.014 g
$LiPF_6$: 7.591 g

Lot# N4P33B

~~1M $LiPF_6$~~
~~(PC:EMC (1:1)~~
1M $LiPF_6$
PC: 1,4-Dioxane

Turned to a gelatinous mass.

PC: 14.695 g
1,4 Dioxane: 17.147 g
$LiPF_6$: 7.6069 g

1M $LiPF_6$
PC:DME (1:1) (250 ml)

PC:DME: Lot# N4P9B & Lot# N4P31A
$LiPF_6$: 38.1282 g

Lot# N4P33C

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Jeffrey A Read
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EXHIBIT B 38/38 HDL

Rate Study of Li-Air in 3 electrolytes (Series A) 4/24/01

Cell	Zr Oxide	Before Discharge (22°C, 751) wt in Air	wt in H_2O	Ch	Rate	After Discharge wt in Air	wt in H_2O	
LA50	28Ω	13.09g	-59.76	1	.05 mA/cm²	13.10g	-42.11g	
LA51	32Ω	12.27g	-74.61	2	.2 mA/cm²	12.26g	-64.71g	
LA52	34Ω	11.60g	-83.91	3	1.0 mA/cm²	11.59g	-80.68g	
LA53	37.5Ω	12.64g	-84.78	4	.05 mA/cm²	12.65g	-65.99g	
LA54	40Ω	12.75g	-124.4	5	.2 mA/cm²	12.75g	-110.87g	
LA55	41Ω	12.23g	-75.8	6	1.0 mA/cm²	12.22	-71.35g	
LA56	52Ω	12.91g	-106.54	20	.05 mA/cm²	12.90	-104.66g	
LA57	47Ω	12.70g	-77.16	21	.2 mA/cm²	11.61	-92.75g	Burst
LA58	46Ω	12.53g	-82.57	22	1.0 mA/cm²	11.49		Burst

5/1/01

Rate Studies of the Li-Air cell are being done to evaluate the effects of various components on the rate behavior. The electrolyte has a strong effect on rate capability and discharge capacity. It is proposed that electrolytes capable of dissolving O_2 should give better rate capability and discharge capacity.

Oxygen solubility has been determined for several common electrolyte solvents, see Solubility Data Series; v.7 Oxygen & Ozone, NIST Call # QD505.A1

Solvent	Solubility	
Propylene Carbonate	1.5×10^{-2} $cm^3 O_2 / cm^3$ solvent	(Pure O_2)
γ-Butyrolactone	5.0×10^{-2} $cm^3 O_2 / cm^3$ solvent	(Pure O_2)
Tetrahydrofuran	22×10^{-2} $cm^3 O_2 / cm^3$ solvent	(Pure O_2)
Tetrahydro-2H-pyran	22×10^{-2} $cm^3 O_2 / cm^3$ solvent	(Pure O_2)
Perfluorobutyltetrahydrofuran	54×10^{-2} $cm^3 O_2 / cm^3$ solvent	(Pure O_2)
Dimethyl Sulfoxide	4.9×10^{-3} $cm^3 O_2 / cm^3$ solvent	(pure O_2)

The solvents are useful in Li-air batteries and should the ones with high oxygen solubility be used, capacity & rate capability should improve.

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